CFTRI-MYSORE
180
Applications of

THE APPLICATIONS OF CHEMICAL ENGINEERING

THE APPLICATIONS OF
CHEMICAL ENGINEERING

Edited by

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PREFACE

This text is a cooperative effort. It was initiated and has been carried to completion by a group of members of the Chemical Engineering Division of the Society for the Promotion of Engineering Education.

The need for a practical laboratory manual in chemical engineering was discussed at the American Institute of Chemical Engineers conference on chemical engineering education in Philadelphia in 1929 and in a succeeding conference in Wilmington in 1935. During the former meeting a number of papers were presented on instructional work in chemical engineering including a paper by the editor of this text which carried the title, "Laboratory Instructions in Chemical Engineering." This discussion was continued at later meetings of the Institute and finally Dr. A. C. Newman was appointed to be the recipient and distributor for such laboratory instructions in chemical engineering as the members of these conferences agreed to submit to him for consideration and distribution.

To the editor's knowledge, this concluded the activities of the American Institute of Chemical Engineers, and the matter was later taken up by the Chemical Engineering Division of the Society for the Promotion of Engineering Education at the Ithaca meeting in 1934 under the direction of Dr. F. C. Vilbrandt. Dr. S. C. Ogburn presented at that meeting a paper entitled, "Chemical Engineering Laboratory Notebooks and Texts" and, as a result of the discussion of this paper, the conference unanimously directed the chairman to appoint a committee on Chemical Engineering Laboratory Directions and Procedure. Dr. Ogburn became chairman of this committee which included the following members: Professor P. M. Horton, Professor W. L. Beuschlein, Professor J. H. James, Professor J. C. Elgin, Professor J. H. Rushton, Professor L. C. Jenness, Professor G. H. Montillon, Professor H. A. Webber, Professor W. N. Cobleigh and Professor H. McCormack.

The initial work of this committee was summarized in a paper presented by Dr. Ogburn at the Atlanta meeting of the Society under the title of "A Study of Chemical Engineering Laboratory Instructions in the United States" and the sessions at Atlanta concluded with the recommendation that at the next conference the subject be "A Symposium on the Laboratory Procedures of Selected Unit Operations." This symposium was held in 1936 at the Madison meeting of the S.P.E.E. and the Division eventually voted that the reports should be edited by an editorial board consisting of Professors McCormack, Hougen and Montillon. At the same time a committee on chemical engineering laboratory instructions was appointed at this meeting, this committee including the following members: H. McCormack, Chairman, Dr. O. A. Hougen, Professor J. H. Rushton, Professor H. T. Ward, Professor R. E. Montonna, Professor J. H. Withrow, Professor J. H. Koffolt, Professor G. H. Montillon, Professor B. F. Ruth, Professor L. T. Work, Professor J. C. Elgin and Professor J. C. Whitwell. Subsequently Professor Montillon and Professor Ruth were for personal and business reasons forced to withdraw from the committee and the work was carried on by the balance of the group under the chairmanship of the editor of this work.

The aim of this committee has been to collect and unify the best available material on the applications of chemical engineering. The committee has had access to the material originally collected by Dr. Ogburn's committee and they have also had access to material submitted to various members of the committee both prior and subsequent to its formation.

This book is in reality a cooperative effort with the co-authors sitting in judgment on the submitted material, using what they deemed wise to use and discarding that which was superfluous or unsatisfactory. However, in the main, material contained in the text is the personal work of the contributing authors and auxiliary material had comparatively little part in the final preparation of the published text.

The basis for the evaluation of the material included in the book has been threefold: satisfactory use in a private laboratory over a period of time; suitability for use with simple equipment that is readily available or that may be constructed without undue difficulty; and, finally, each experiment is definitely related to the theoretical presentation of chemical engineering and possible of accomplishment in a single four-hour laboratory period. The editor and the contributing authors sincerely hope that these objectives have been well met and that the book contains a judiciously selected and well organized list of experiments. Seventy-five applications are given in order that the teacher may select those best adapted for his purposes and for his available equipment; possibly one-third of this number is sufficient for the average course.

Certain unit operations probably have an excess of applications pertaining thereto; other unit operations are perhaps too brief and will require amplification by the teacher. This unbalance is due to the ready availability of material on certain unit operations and the lack of adequate material as regards others.

The editor wishes to express deep appreciation and satisfaction with the work that has been done by the contributing authors and also to express his gratitude to the membership of the Division of the Society for the Promotion of Engineering Education which inaugurated and sponsored the undertaking which has culminated in the publication of this text. The editor and his co-authors sincerely believe that this activity is a worthwhile contribution to the advancement of chemical engineering education.

June, 1940.

THE EDITOR.

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THE APPLICATIONS OF CHEMICAL ENGINEERING

CHAPTER I

THE MEASUREMENT OF TEMPERATURE

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These experiments are based on mimeographed laboratory direction sheets from Armour Institute of Technology, prepared by Prof. H. McCormack, and from the University of Wisconsin, prepared by Dr. K. M. Watson and Prof. R. A. Ragatz.

TEMPERATURE SCALES

Two temperature scales are in common use in the United States at the present time, the Centigrade scale and the Fahrenheit scale. The Centigrade scale is usually employed in scientific work, while the Fahrenheit scale is usually preferred for industrial work.

On the Centigrade scale, the temperature of melting ice at 1 atmosphere pressure (760 mm. of mercury at the temperature of melting ice) is assigned a value of zero degrees, while the temperature of steam condensing under 1 atmosphere of pressure is assigned a value of 100 degrees.

On the Fahrenheit scale, the temperature of melting ice at 1 atmosphere pressure is assigned a value of 32 degrees, while the temperature of steam condensing under 1 atmosphere of pressure is assigned a value of 212 degrees.

Conversion of temperatures from the Fahrenheit to the Centigrade scale and vice versa is accomplished by the following relationships, where C represents Centigrade temperature and F represents Fahrenheit temperature.

$$C = 5/9\ (F - 32)$$
$$F = 9/5\ (C) + 32$$

In performing many calculations it is necessary to convert Centigrade or Fahrenheit temperatures into absolute temperatures. Careful measurements have shown that the absolute zero lies 273.2 Centigrade degrees below the melting point of ice. Therefore, on the *absolute* Centigrade scale, the temperature of melting ice will be 273.2 degrees and the temperature of condensing steam at 1 atmosphere pressure will be 373.2 degrees. Absolute Centigrade temperatures are conventionally designated by the letter K in honor of the English physicist Lord Kelvin.

$$K = C + 273.2$$

A temperature may be converted into absolute Fahrenheit degrees rather than absolute Centigrade degrees if desired. Since any specific temperature interval contains 1.8 as many Fahrenheit as Centigrade degrees (from the freezing point to the boiling point of water we have 180 Fahrenheit degrees and 100 Centigrade degrees), absolute zero on the Fahrenheit scale must lie 273.2 × 1.8 or 491.8 Fahrenheit degrees below the freezing point of water. Since the freezing point of water is assigned a value of 32 on the Fahrenheit scale, the absolute zero must be 491.8 − 32 or 459.8 degrees below the zero point on the Fahrenheit scale. To convert a Fahrenheit temperature to absolute Fahrenheit degrees, it is only necessary to add 459.8. Absolute Fahrenheit temperatures are conventionally designated by the letter R in honor of the engineer Rankine, who made important contributions to the theory of heat engines.

$$R = F + 459.8$$

Conversion of Kelvin degrees into Rankine degrees and vice versa is accomplished by the following equation:

$$R = 9/5\,K$$

The following tabulation shows the Centigrade, Fahrenheit, Kelvin, and Rankine temperatures at three important fixed points: the absolute zero, melting ice at 1 atmosphere pressure, and condensing steam at 1 atmosphere pressure.

Fixed Point	Temperatures			
	Centigrade Scale	Fahrenheit Scale	Kelvin Scale	Rankine Scale
Condensing steam at 1 atmosphere pressure	100° C.	212° F.	373.2° K.	671.8° R.
Melting ice at 1 atmosphere pressure	0° C.	32° F.	273.2° K.	491.8° R.
Absolute zero	−273.2° C.	−459.8° F.	0° K.	0° R.

The reference standard for temperature measurements is the constant volume gas thermometer, which ordinarily utilizes either helium, hydrogen, or nitrogen as the thermometric substance. The instrument operates on the well-known principle that if a gas is confined at constant volume, the pressure will increase if the temperature is increased. If behavior of the gas used as a thermometric substance were ideal, absolute temperatures computed on the assumption of a direct proportionality existing between absolute temperature and pressure would conform exactly to the ideal thermodynamic temperature scale on which, according to Kelvin, "the absolute values of two temperatures are to one another in the proportion of the heat taken in to the heat rejected in a reversible thermodynamic engine working with a source and refrigerator at the higher and lower of the temperatures, respectively." Actually, neither helium, hydrogen, nor nitrogen exhibit absolutely ideal behavior. However, for each gas the departure from ideal behavior is known, and therefore temperatures computed on the assumption of direct proportionality between absolute temperature and pressure may be properly corrected for departure of the gas from ideal behavior, and thus the true thermodynamic temperature may be evaluated from the pressure reading of the thermometer.

The most important use of the constant volume gas thermometer has been in the determination of temperatures corresponding to various fixed points such as the freezing and boiling points of pure substances. The purpose of these investigations has been to provide temperature values for easily reproducible fixed points which could then be used to calibrate various temperature measuring devices. As in all experimental work, the temperature values reported by different investigators were not in perfect agreement. It therefore was desirable that a committee with international representation consider the various temperature values reported for the fixed points and that an agreement be reached as to the best values to use. This work was started as far back as 1911, but did not result in a general agreement until 1927, when the Seventh General Conference of Weights and Measures adopted the so-called International Temperature Scale.[1] The following numerical values were assigned to fixed points used in the calibration of temperature measuring devices.

In those instances where one of the two phases involved in the equilibrium at the fixed point is a gas, variations in barometric pressure will affect the temperature. Accordingly, the following equations are provided for applying a correction for barometric pressure.

p = pressure in mm. mercury.

t_p = *Centigrade* temperature, at pressure p.

Oxygen

$$t_p = -182.97 + 0.0126(p - 760) - 0.0000065(p - 760)^2$$

---

[1] The International Temperature Scale, Burgess, Research Paper No. 22, U. S. Bureau of Standards. Also published in Bureau of Standards Journal of Research, Oct. 1928.

TABLE 1

FIXED POINTS FOR THE INTERNATIONAL TEMPERATURE SCALE

Pressure = 1 standard atmosphere in each instance

BASIC FIXED POINTS

	Deg. Cent.	Deg. Fahr.
1. Temperature of equilibrium between liquid and gaseous oxygen	−182.97	−297.35
2. Temperature of melting ice in contact with air-saturated water (by definition)	0.000	32.000
3. Temperature of condensing steam (by definition)	100.000	212.000
4. Temperature of condensing sulfur vapor	444.60	832.28
5. Temperature of freezing silver	960.5	1760.9
6. Temperature of freezing gold	1063	1945

SECONDARY FIXED POINTS

	Deg. Cent.	Deg. Fahr.
1. Temperature of equilibrium between solid and gaseous carbon dioxide	−78.5	−109.3
2. Temperature of freezing mercury	−38.87	−37.97
3. Transition point of sodium sulfate $Na_2SO_4 \cdot 10H_2O \rightleftarrows Na_2SO_4 + 10H_2O$	32.38	90.28
4. Temperature of condensing naphthalene vapor	217.96	424.33
5. Temperature of freezing tin	231.85	449.33
6. Temperature of condensing benzophenone vapor	305.9	582.6
7. Temperature of freezing cadmium	320.9	609.6
8. Temperature of freezing lead	327.3	621.1
9. Temperature of freezing zinc	419.45	787.01
10. Temperature of freezing antimony	630.5	1166.9
11. Temperature of freezing copper in a reducing atmosphere	1083	1981
12. Temperature of freezing palladium	1555	2831
13. Temperature of melting tungsten	3400	6152

Steam

$$t_p = 100.000 + 0.0367(p - 760) - 0.000023(p - 760)^2$$

Sulfur

$$t_p = 444.60 + 0.0909(p - 760) - 0.000048(p - 760)^2$$

Carbon Dioxide

$$t_p = -78.5 + 0.1443(t_p + 273.2)\log_{10}\left(\frac{p}{760}\right)$$

Naphthalene

$$t_p = 217.96 + 0.208(t_p + 273.2)\log_{10}\left(\frac{p}{760}\right)$$

Benzophenone

$$t_p = 305.9 + 0.194(t_p + 273.2)\log_{10}\left(\frac{p}{760}\right)$$

All of the above equations are of the general form

$$t_p = t_{760} + \text{Correction Term}$$

In case calculations are to be carried out on the basis of Fahrenheit temperatures, it is suggested that the "Correction Term" be evaluated in Centigrade degrees, utilizing the above equations. The value thus obtained, when multiplied by 1.8, yields the correction in Fahrenheit degrees, and this then may be added to the Fahrenheit temperature corresponding to 1 atmosphere of pressure, as given in Table I.

While the temperature values adopted for the basic fixed points selected to define the International Temperature Scale were based upon researches with the constant volume gas thermometer, and represent temperatures on the thermodynamic scale, the committee took cognizance of the fact that the constant volume gas thermometer is an extremely difficult instrument to use, and that other temperature measuring devices were capable of a much greater degree of precision. Accordingly, the resolution adopted as defining the International Temperature Scale not only assigned numerical values to the basic fixed points (based on researches with the constant volume gas thermometer), but also divided the temperature scale up into several ranges and specified the temperature measuring device that is to be considered as defining the scale between the basic fixed points in each range. In brief, from −190° C. to 660° C., the scale between basic fixed points is defined by the platinum resistance thermometer, from 660° C. to 1063° C., the scale between basic fixed points is defined by the platinum vs. 90% platinum + 10% rhodium thermocouple, while above 1063° C., the scale is defined by an optical pyrometer utilizing Wien's law of radiation with the melting temperature of gold as a reference point. The secondary fixed points given in Table I were determined by utilizing the temperature measuring devices specified as standard in each temperature range.

In highly precise investigational work, where exact agreement with the International Temperature Scale is demanded, it is necessary to adhere rigidly in each temperature range to the use of those instruments specified as defining the scale. Furthermore, calibration of these instruments must be made at the primary, rather than at the secondary fixed points. Usually, however, such extreme accuracy is not demanded and it is very common practice to use temperature measuring devices which are not used at all as defining standards for the International Temperature Scale. Furthermore, temperature measuring devices which are used as defining standards for the International Temperature Scale are very frequently employed at temperatures outside the range of temperature wherein they serve to define the temperature scale. In all instances except those where exact agreement with the International Temperature

Scale is demanded, any conveniently located fixed points, either primary or secondary, may be used for calibration purposes.

At this point, it will be desirable to give a brief description of the more important types of temperature measuring devices.

MERCURY IN GLASS THERMOMETER

The most commonly used temperature measuring device is the mercury in glass thermometer. This instrument when manufactured with care and properly calibrated may have an accuracy of 0.005° C. It is a fragile instrument and at its best does not possess the accuracy of a resistance thermometer. Then, too, the range of temperature through which it may be used (−35° C. to 550° C.) is small compared with the complete range through which temperature measurements are desired in the industrial operations of the present. The temperatures which we may wish to measure may be as low as −260° C. or as high as 2000° C. It is to be noted, therefore, that even were the mercury in glass thermometer an ideal instrument, additional temperature measuring devices would be necessary. A method is many times desired by which the temperatures occurring during a certain elapsed period may be recorded, and this is not feasible with the mercury in glass thermometer. These demands have led to the development of various types of temperature measuring and recording instruments.

ELECTRICAL RESISTANCE THERMOMETERS

One of the most accurate types of temperature measuring instruments is the resistance thermometer, in which the variation in the electrical resistance of a coil of wire serves as a means of evaluating temperature. Siemens first suggested the use of this property as an accurate means of temperature determination as early at 1871, but due to an incomplete knowledge of the factors which might cause a variation in the circuit resistance other than that produced by temperature change, the method received scant attention for about thirty years. Later investigators showed that the earlier difficulties resulted primarily from faulty protection of the resistance coils and that when the resistance coils were properly protected, the method was an extremely accurate one.

There are two metals finding most general use in making resistance coils for resistance thermometers, pure platinum for temperatures up to 1000° C. and pure nickel for temperatures below 250° C.

The details of construction that should be observed if the resistance thermometer is to give reliable results may be illustrated by discussing the construction of a platinum resistance thermometer. The coil of platinum wire is wound on two strips of mica set crosswise. These coils of wire are held in place by notches in the mica strips. The coil is insulated from its sheath by strips of mica somewhat thinner and wider than those on which it is wound. Before winding, the coil is annealed by heating to a temperature of from 1000° to 1200° C. by passing an electric current through it. After winding and attaching the leads so that no further handling is necessary, the coil is carefully washed in distilled water and then heated to a bright red to drive off all volatile matter and to re-anneal the wire.

Compensation for lead wire resistance is usually accomplished by supplying two dummy leads, which are of exactly the same size and length as the two leads to the coil, and which run parallel to the coil leads. Thus, the dummy leads have exactly the same resistance as the leads to the coil, and if they are inserted into the Wheatstone bridge circuit in the arm adjacent to that containing the coil, the effect of lead wire resistance is cancelled out of the measurement.

THERMOELECTRIC PYROMETERS

The operation of the thermocouple depends upon the fact that in a closed circuit composed of two metals an electromotive force will be set up if the two junctions are at different temperatures. The intensity of the electromotive force produced is dependent upon the hot and cold junction temperatures, and the nature of the two metals comprising the circuit. Different combinations of metals vary in the electromotive force produced per unit change in temperature. The characteristics of the specific couple must be known, therefore, in order to interpret the reading in millivolts into its equivalent temperature.

It is obvious that it will be desirable to select such combinations of metals as will produce a high electromotive force per unit temperature change, if the characteristics of the metals are otherwise satisfactory. Almost any selection of two dissimilar metals might be employed as far as the production of an electromotive force is concerned. Practically, however, only a few combinations of metals have been found which possess all or nearly all of the desired characteristics. A thermocouple, in addition to developing a relatively large electromotive force, should possess the ability to resist corrosion or oxidation and the temperature-electromotive force relation should be such that the latter increases continuously with increasing temperature over the range to be employed.

The following thermocouple combinations have come into widespread use, as they fulfill the specifications set forth above to a greater degree than do other possible combinations.

	Upper Limit	
	Deg. C.	Deg. Fahr. (Rounded Off Values)
Platinum vs. 90% platinum + 10% rhodium	1500° C.	2730° F.
Chromel vs. Alumel	1100° C.	2010° F.
Iron vs. Constantan	900° C.	1650° F.
Copper vs. Constantan	360° C.	680° F.

One of the early combinations of metals to come into general use was the platinum versus 90% platinum + 10% rhodium couple. It is a very satisfactory couple, but has three objectionable characteristics. The materials are very expensive, the electromotive force per unit of temperature change is rather small, and platinum will alloy with nearly any common metal in the molten condition. Because of the expense of the material, the diameter of wire employed in these couples is small, a common diameter being 0.6 mm. Frequently couples with wire of 0.4 mm. diameter, requiring but half as much metal, are used.

The platinum versus 90% platinum + 10% rhodium combination possesses one desirable feature to a greater extent than any other couple. Different manufacturers can duplicate their couples year after year so that not only are different couples of one manufacturer interchangeable on industrial direct reading instruments, but carefully made couples from another maker will function equally well. Data available on this characteristic show that the variation from year to year from different manufacturers will not be greater than 2° to 3° C.

The expense of the platinum versus 90% platinum + 10% rhodium couple along with its property of readily forming alloys led to a search for other combinations of metals possessing satisfactory thermoelectric properties. The iron-constantan and copper-constantan pairs were among the earlier types of base metal couples used. These couples are still employed and function satisfactorily within certain temperature ranges.

Later combinations have been found which function satisfactorily through a wider temperature range and do not deteriorate as rapidly as the iron-constantan and copper-constantan couples. The most satisfactory base-metal combination developed to date is the Chromel-Alumel thermocouple. This couple may be used in continuous service up to 1100° C., has excellent resistance to oxidation, and retains its calibration well unless used under conditions under which it undergoes contamination.

A difficulty encountered in the early manufacture of the base metal alloy couples was the inability of the manufacturer to reproduce the same e.m.f.—temperature relation from one casting to another. This was a source of considerable trouble in the pioneer days of base metal couples. Manufacturers today, however, have developed their operations and skill to the point where this trouble has been largely eliminated. Couples are now generally interchangeable on the reading instrument when both are from the same maker.

The material used in the production of any of these alloy couples is so inexpensive that the couple may be made in any size wire desired and of any length required. The customary wire sizes are numbers 6 and 8 B. & S. gage, and the couples usually vary from two to twenty feet in length.

The reading instruments used with thermocouples are of two types: millivoltmeters operating on the principle of the movable coil galvanometer, and potentiometers. The requirements of a millivoltmeter for industrial practice are about as follows. It should be dust proof, of sufficient deflection range, and at the same time should have an open scale which is well marked and easily read. The deflection of the galvanometer should be periodic or deadbeat, and the open circuit reading should remain constant even after large deflections, long maintained. There should be a suitable leveling device which has been accurately adjusted and the case and other supports should be non-warping. The zero position of the pointer should be readily adjustable.

In the potentiometer, an entirely different principle of operation is employed than in the millivoltmeter. The electromotive force of the thermocouple is balanced by impressing a variable and known e.m.f. across the terminals of the thermocouple. When a galvanometer which is placed in series with the thermocouple reads zero, balance is achieved, and the thermocouple e.m.f. exactly equals the e.m.f. impressed across the terminals of the thermocouple. Inasmuch as balance is attained with no current flowing in the couple, the reading is unaffected by the resistance of the thermocouple. This constitutes the great advantage of the potentiometric method of measuring thermocouple e.m.f.s over the millivoltmeter method.

RADIATION PYROMETERS

The types of pyrometers thus far described reach their upper temperature limits under 1500° C. and in each of them some portion of the instrument must be directly in contact with the space or material whose temperature is being measured. These demands are not encountered in radiation pyrometry, as this instrument may be constructed and calibrated to measure any obtainable temperature and no part of the instrument need be in contact with the space or material under observation.

The earliest suggestion of the simple relationship between the radiation emitted from a surface and its temperature was made by Stefan in 1879.

From a limited number of observations, he concluded that the total energy emission from a body is proportional to the fourth power of the temperature expressed in degrees Kelvin. Subsequent investigators found that this generalization was valid only in the case of a "black body," that is, a body which has the ability to absorb all radiation incident upon it, and which has a maximum capacity to emit radiation.

Metallic surfaces with high reflecting power deviate very noticeably from Stefan's generalization. Boltzmann, in 1884, stated that the relationship expressed by Stefan is valid only for materials of the ideal "black body" type, and this remains our conception of the relationship between radiation and temperature at the present time.

Pyrometers based on the fourth power law used for the measurement of higher temperatures are merely thermocouples so arranged that the instruments are direct reading, rugged, correct in action, and so constructed as to render the readings independent of the distance between the pyrometer and the hot body. The latter provision holds true only within certain limits.

Fery appears to have been the first to construct a temperature measuring instrument based on the Stefan-Boltzmann law. This instrument was capable of measuring temperatures between 500° C. and 1500° C. and like all earlier types was so constructed as to require focusing on the object the temperature of which was to be measured. Later instruments have eliminated the necessity for focusing.

The radiation pyrometer, since it uses a thermocouple as the actual temperature measuring device, may be used with the same types of indicating instruments as have already been discussed in conjunction with thermocouples. Usually, however, the manufacturer supplies a millivoltmeter, graduated to read source temperature directly.

OPTICAL PYROMETERS

It is possible to construct a temperature measuring device where luminous radiations are measured instead of the total radiant energy as is done in radiation pyrometry.

Usually, the temperature is determined by measuring the intensity of the luminous radiation at some particular wave length, quite commonly in the red end of the spectrum at about 65 microns.

At the present time there is but one widely used type of optical pyrometer, the disappearing filament type. The original disappearing filament type was invented by Morse. Several refinements in the instrument were later introduced by Holburn and Kurlbaum. The filament of a small electric lamp is located in a telescope tube which is equipped with an objective lens and an ocular. The ocular is focused sharply on the lamp filament. Then the telescope is directed at the object whose

TABLE 2

Classification of Industrial Temperature Measuring Instruments *

I. Contact Type

The temperature measuring device is in contact with the body whose temperature is being measured.

		Useful Range of Operation	
		Centigrade	Fahrenheit
1. Expansion Thermometers			
(a) *Expansion of Gases*			
Pressure:	Nitrogen	−130 to 540° C.	−200 to 1000° F.
(b) *Expansion of Liquids*			
Liquid in Glass:	Mercury	− 30 to 540° C.	− 30 to 1000° F.
	Ethyl alcohol	− 70 to 120° C.	−100 to 250° F.
	Pentane	−180 to 20° C.	−300 to 70° F.
Liquid Pressure:	Methyl alcohol	− 50 to 70° C.	− 60 to 150° F.
	Xylene	− 40 to 400° C.	− 40 to 750° F.
	Mercury	− 40 to 540° C.	− 40 to 1000° F.
	Methyl chloride	− 30 to 40° C.	− 30 to 100° F.
	Iso-butane	− 20 to 35° C.	− 5 to 95° F.
	Normal butane	− 10 to 50° C.	15 to 120° F.
	Ethyl chloride	0 to 65° C.	30 to 150° F.
	Heavy hydrocarbon oil	10 to 150° C.	40 to 300° F.
	Ether	20 to 90° C.	70 to 195° F.
	Acetone	40 to 110° C.	110 to 235° F.
	Ethyl alcohol	70 to 130° C.	155 to 260° F.
	Butyl alcohol	100 to 180° C.	220 to 345° F.
	Aniline	160 to 240° C.	320 to 465° F.
(c) *Vapor Pressure:*	Sulfur dioxide	− 30 to 120° C.	− 20 to 250° F.
	Ether	10 to 150° C.	50 to 300° F.
	Ethyl alcohol	65 to 200° C.	150 to 400° F.
(d) *Differential Expansion of Solids*			
Bimetallic Bar		25 to 70° C.	80 to 160° F.
Silica and Monel Metal		90 to 650° C.	200 to 1200° F.
2. Electrical Resistance Thermometers			
(a) *Base Metal*			
Copper		−180 to 120° C.	−300 to 250° F.
Nickel		−180 to 120° C.	−300 to 250° F.
(b) *Noble Metal*			
Platinum		−180 to 1000° C.	−300 to 1800° F.
3. Thermoelectric Pyrometers			
(a) *Base Metal*			
Copper—Constantan		−200 to 360° C.	−325 to 700° F.
Iron—Constantan		0 to 900° C.	32 to 1650° F.
Chromel—Alumel		0 to 1100° C.	32 to 2000° F.
(b) *Noble Metal*			
Platinum vs. 90% Platinum + 10% Rhodium		0 to 1500° C.	32 to 2700° F.
Platinum vs. 87% Platinum + 13% Rhodium		0 to 1500° C.	32 to 2700° F.

II. Distance Type

The temperature measuring device is not in contact with the body whose temperature is being measured.

1. Radiation Pyrometers	Over 500° C.	Over 950° F.
2. Optical Pyrometers	Over 600° C.	Over 1100° F.

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* Adapted from tabulation by Rimbach in *Instruments*, Vol. 1, p. 441 (1928).

temperature is to be measured, and the image brought into focus by moving the objective lens back and forth. Thus the image of the source is superimposed upon the image of the lamp filament. High transmission red glass is placed over the eyepiece so that approximately monochromatic illumination is assured. The lamp circuit contains a rheostat by means of which the current to the lamp may be regulated until the tip of the lamp filament has the same brightness as the source viewed. The current passing is then read on an ammeter and the corresponding temperature is obtained from the plot or table of current versus temperature. It is necessary to determine experimentally the relationship between current flowing and temperature at a minimum of three, and preferably more, standardization points. Unless the lamps are operated at too high a temperature, their calibration is good for several hundred hours of ordinary use.

The instruments of today are usually equipped with a dark glass having a certain definite absorptive power which may be interposed if desired between the object whose temperature is required and the lamp filament of the instrument. Without this absorption glass, temperatures up to 1400° C. can be measured and with it the range is increased to 2800° C. The measuring instrument used with this pyrometer is an ammeter adapted to the current range covered in the normal operation of the instrument.

In using an optical pyrometer, due recognition must be taken of the fact that different bodies at the same temperature, when radiating in the open, will emit radiation of different intensity. Polished metals, for example, when radiating in the open, will appear far less bright than a substance such as carbon at the same temperature. Fortunately, all bodies when placed within a uniformly heated enclosure will appear of equal brilliance when they attain the temperature of the enclosure, and the radiation given off under those conditions equals that of the ideal "black body" or perfect radiator. Optical pyrometers are always calibrated against black body radiation. Hence, an optical pyrometer when sighted within a uniformly heated enclosure will indicate true temperature. If, however, the pyrometer is sighted on a non-black body radiating in the open (molten metal being tapped into a ladle, for example) the instrument will read low, and a correction must be applied to the indicated temperature.

EXPERIMENT NO. 1

THE WHEATSTONE BRIDGE AND THE POTENTIOMETER

For the measurement of direct current resistances, potentials, and currents, the Wheatstone bridge and the potentiometer are the standard precision instruments. Their sensitivities may be made very high and yet a single instrument may be used over a wide range of measurements. The accuracy of neither instrument is dependent on such questionable constants as the field of a permanent magnet, the tension of a spring or the friction of a bearing.

Object. To construct and study elementary forms of a modified Wheatstone bridge and a potentiometer and to study their operation and uses.

THE WHEATSTONE BRIDGE

One of the most useful and practical forms of the Wheatstone bridge is that employing a calibrated slide wire. However, for pyrometric work it is desirable that the fixed resistance arms of the bridge bear the constant ratio of 1.0 to each other. For this reason it is necessary to modify somewhat the familiar Wheatstone bridge circuit when adapting it to pyrometric practice. On the accompanying diagram in Figure 1 is shown this modified circuit as generally used. In this figure, (M) and (N) are resistances of equal magnitude forming arms (1) and (2) of the bridge. Resistance (D) is a calibrated slide wire on which is the movable contact (E). Resistance (C) is of a magnitude exactly equal to the total value of (D). Resistance (X) represents the unknown whose value it is desired to measure. With the battery and galvanometer (G) connected as shown, the arms of the bridge are indicated by brackets and numerals. Since $(M) = (N)$, it is apparent that when the bridge is in balance the total resistance in arm 3 must equal that in arm 4. If (y) is the resistance of that portion of the slide wire (D) which lies to the right of contact (E), the total resistance in arm 3 is equal to $y + x$. The total resistance in arm 4 is equal to $C + D - y$. When the bridge is balanced the unknown resistance X then becomes equal to $2(D - y)$.

With such a bridge, resistances may be measured to a maximum value equal to 2D.

Procedure. *Part 1. Measuring Electrical Resistances.* A bridge is set up as shown in Figure 2.

The resistance of slide wire (D) and of resistance coil (C) should be obtained from the instructor. The two resistances should be practically identical.

Two resistance coils "A" and "B" are obtained from the instructor, and their resistances measured by means of the Wheatstone bridge. In bringing the bridge into balance, a preliminary balance is made with the short circuiting switch (S) open, so that the protective resistance is effective in protecting the galvanometer from unduly large currents. Furthermore, the key on the slide wire which closes the battery circuit should be closed only momentarily by a light tap in order to prevent too violent a deflection of the galvanometer. After the preliminary adjustment has been made, final balance is secured by short circuiting the protective resistance by closing the switch (S).

When balance is secured as indicated by a zero reading of the galvanometer, the slide wire scale reading is recorded. For each coil, at least three balances should be made, and the average values used in the calculations.

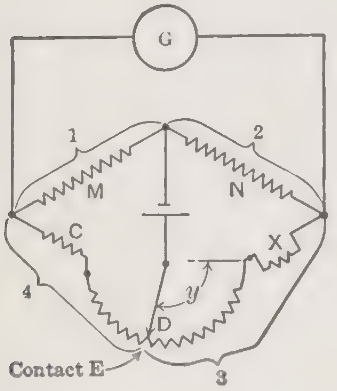

FIG. 1

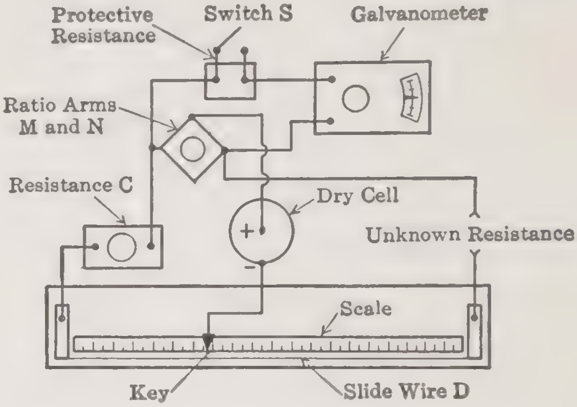

FIG. 2

After a satisfactory set of measurements has been made, the resistance of coil "A" and of coil "B" are to be calculated.

THE POTENTIOMETER

The theory of operation of a potentiometer of the null or balanced type is illustrated by Figure 3. A dry cell sends current through the slide wire ABC. A fall of potential therefore exists along the slide wire. In parallel with the slide wire is a shunt that contains the unknown potential. One end of this shunt circuit is permanently attached to the left end of the slide wire at (A), while the other end (B) is adjustable

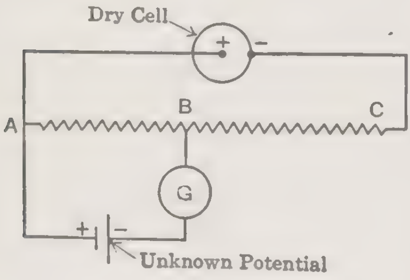

FIG. 3

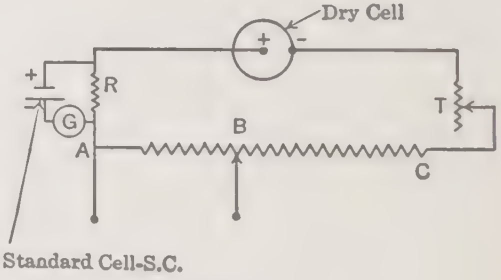

FIG. 4

to any position along the slide wire. The shunt circuit also has a galvanometer (G) in series with the unknown e.m.f. The unknown e.m.f. is put into the circuit in such a manner that it is in opposition with respect to the potential difference that exists between (A) and (B) on the slide wire (+ to + and − to −). Unless contact (B) happens by chance to be at the exact point of balance, the unknown e.m.f. will not be of exactly the same magnitude as the potential difference between (A) and (B), consequently a small current will flow in the shunt circuit, and the galvanometer will show a deflection. If now the adjustable contact (B) is moved back and forth on the slide wire, a point will be found at which the galvanometer (G) shows zero deflection. This signifies that no current is flowing in the shunt circuit (current, however, is flowing in the slide wire at all times), from which it is concluded that the unknown e.m.f. is exactly balanced by the potential difference between (A) and (B). To summarize, when the instrument is in balance (zero deflection of the galvanometer G), the unknown e.m.f. exactly equals the potential difference between (A) and (B) on the slide wire.

The problem of evaluating the numerical magnitude of the unknown e.m.f. is thus resolved into determining the potential difference between the fixed point (A) and the movable contact (B) of the slide wire. This requires: (a) that we know the resistance between (A) and (B), and (b) that we know the current flowing in the slide wire.

In constructing a potentiometer, a slide wire is always used that has been carefully calibrated with respect to resistance. Consequently, for any particular setting of the adjustable contact (B), the resistance between (A) and (B) is known. With respect to evaluating the current flowing in the slide wire, the first method that suggests itself is the use of an ammeter in series with the dry cell and the slide wire. However, such a method would have the following disadvantages:

(1) The current through the slide wire must be low to prevent heating (which would alter the slide wire resistance), consequently a very sensitive ammeter would be required.

(2) With an ordinary type of milliammeter, the percentage accuracy of the current measurement, and consequently of the e.m.f. measurement, is considerably less than for the method described below.

(3) If an ammeter is used, the accuracy of the potential determination is dependent on the accuracy of the ammeter, which in turn depends on the constancy of the electromagnets, of the frictional resistance in the bearings and of the resisting torque of the coiled spring or suspension wires.

A most satisfactory method is available by which the current may always be adjusted to the same, definitely known value. Small galvanic cells are manufactured which maintain a voltage constant to within less than 0.1% if no appreciable current is withdrawn from them.

Figure 4 shows such a standard cell (S.C.) connected across a known, fixed resistance (R) which is in series with the battery circuit. In series with the cell is a galvanometer (G). *The polarity of the standard cell must be such that its voltage opposes the voltage drop produced by the current flowing through (R) from the other battery.* If now the current through (R) is properly adjusted by means of a rheostat (T) the voltage drop across (R) may be made to equal exactly the voltage of the standard cell. Under such conditions the galvanometer (G) will show no deflection and the current through (R) is equal to $\frac{\text{e.m.f. of standard cell}}{\text{resistance of coil (R)}}$. With the current in the potentiometer adjusted to this known value, the potential between (A) and (B) at any setting is readily calculated from the known resistances. Since the current will always be adjusted to exactly this same value the potentials corresponding to a particular setting of (B) will always be the same and the scale may be marked to read potentials directly. This comprises a complete potentiometer.

In using the instrument to measure a potential the current is first adjusted to the correct value by connecting the standard cell as in Figure 4. The standard cell may then be disconnected. The unknown potential is connected across (A) and (B) *with its polarity such as to oppose the voltage drop in the potentiometer.* A galvanometer is connected in series with the unknown potential. Contact (B) is then moved about until the potentials are exactly balanced as indicated by the zero reading of the galvanometer. The potential between (A) and (B) may then be calculated from the constant current value and the resistance between (A) and (B).

It is apparent that the potentiometer can *directly* measure only potentials. However, the instrument is the most generally useful of all electrical measuring devices and may also be used to measure current and resistance by indirect but highly accurate methods. The value of a current is determined by allowing it to pass through a resistance of known magnitude and using the potentiometer to measure the potential drop produced. The value of the current is calculated from Ohm's law. The magnitude of a resistance is measured by connecting it in series with a standard resistance and passing the same current through both. The potential drop across each is measured with the potentiometer. The values of the two resistances then are to each other as the potential drops across them.

Part 2. Adjusting the Battery Current. The slide wire previously used as a Wheatstone bridge is connected to form a potentiometer as shown in Figure 5. For resistance (R), the resistance "A" is used whose value was determined on the bridge.

The rheostat (T) is then adjusted until, when the key is depressed, the galvanometer shows no deflection.

It is extremely important that the connections be made so that the e.m.f. of the standard cell is in opposition to the dry cell e.m.f. Otherwise it is impossible to bring the instrument into balance. The polarities as indicated on the wiring diagrams should be carefully observed and followed.

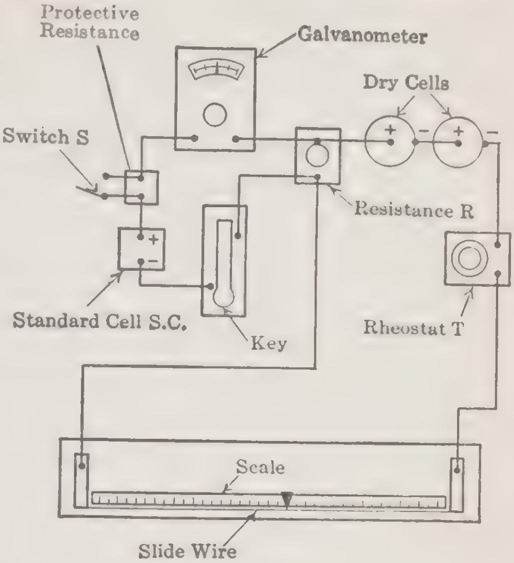

FIG. 5

In bringing the instrument into balance (zero reading of the galvanometer), preliminary adjustment should always be made with the short circuiting switch (S) open, in order that the protective resistance will protect the galvanometer from unduly large currents. Furthermore, the key closing the galvanometer circuit should always be tapped and not held down continuously. Final balance should be secured with the short circuiting switch (S) closed.

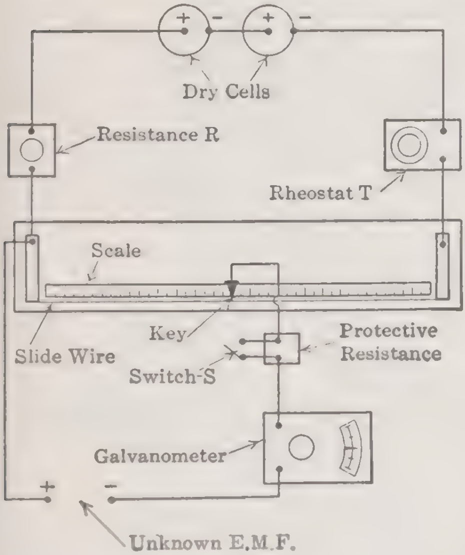

FIG. 6

From the known voltage of the standard cell, and the resistance of coil "A" as determined in Part 1, the current flowing through the potentiometer slide wire is to be computed.

Part 3. Measuring an Unknown Potential. Without disturbing the setting of the rheostat (T), the standard cell, key, and galvanometer are disconnected. (Fig. 6.) The galvanometer is connected into the circuit as shown in Figure 6. The instrument is now used to measure the voltage of a galvanic cell, supplied by the instructor. The cell has electrodes of copper (−) and carbon (+), and the electrolyte is an aqueous solution containing 10% NaCl. The cell is connected as the unknown potential and the contact on the slide wire moved until, on depressing it, the galvanometer shows no deflection. The voltage of the unknown cell is then calculated from the setting of the contact, the resistance of the slide wire, and the current flowing in it.

Part 4. Measuring Current with a Potentiometer. The potentiometer is then used to determine the current flowing through an ordinary incandescent lamp. The lamp is connected across a source of 110 volt *D.C.* in series with resistance "B" whose magnitude was previously determined with the Wheatstone bridge. Consult the accompanying wiring diagram. (Fig. 7.) Particular care must be taken to have all polarities correct.

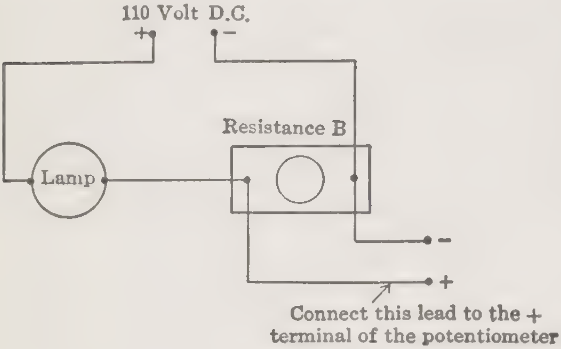

Fig. 7

The potential drop across the resistance is then measured with the potentiometer. From a knowledge of the potential drop across the coil, and the coil resistance, the current flowing through the coil and lamp may be computed.

Part 5. Measuring Resistance with a Potentiometer. The magnitude of resistance "C" which was used in the construction of the Wheatstone bridge is now checked with the potentiometer. The unknown resistance "C" is connected in series with the known resistance "B," directly across the terminals of a dry cell as shown on the wiring diagram. The potential drop across each is measured with the potentiometer. The resistance of "C" is then calculated.

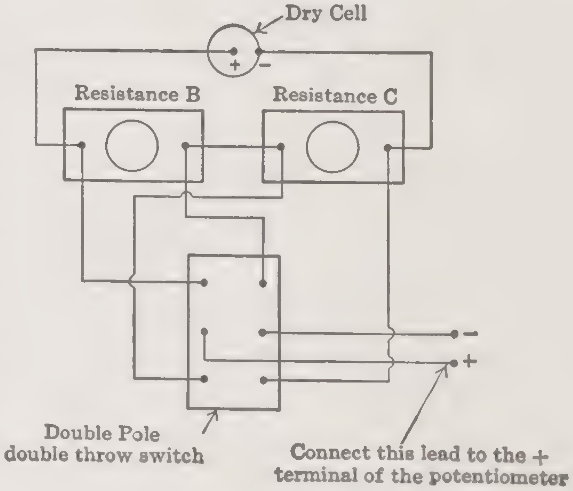
Dry Cell
+
−
Resistance B
Resistance C
−
+
Double Pole double throw switch
Connect this lead to the + terminal of the potentiometer

Fig. 8

If all measurements were carefully made in this experiment, the resistance of the ‘C" coil as measured in Part 5 should be very nearly equal to the resistance value obtained from the instructor in Part 1.

REPORT

1. A calculation sheet is to be presented giving all computations called for in this experiment.

SAMPLE DATA AND CALCULATIONS

Part 1. Measurement of Resistance:

Bridge Constants

"C" coil: 3.080 ohms
Slide wire: 3.081 ohms

Resistance of "A" Coil

Distance from left end of scale to contact point when bridge is in balance = 83.10 cm. (Ave. of 3 readings)

$$\text{Resistance of "A" coil} = 2(D - y) = 2 \times \frac{83.10}{100.00} \times 3.081 = 5.120 \text{ ohms}$$

Resistance of "B" Coil

Distance from left end of scale to contact point when bridge is in balance = 74.52 cm. (Ave. of 3 readings)

$$\text{Resistance of "B" coil} = 2(D - y) = 2 \times \frac{74.52}{100.00} \times 3.081 = 4.592 \text{ ohms.}$$

Part 2. Current Through Potentiometer Slide Wire:

Voltage of standard cell = 1.55 volts
Resistance of "A" coil (see Part 1) = 5.12 ohms.

$$\text{Current through slide wire} = \frac{1.55}{5.12} = 0.303 \text{ ampere.}$$

Part 3. Voltage of Electrolytic Cell:

Distance from left end of scale to contact point when potentiometer is in balance = 25.9 cm. (Ave. of 3 readings)
Total voltage across slide wire = $RI = 3.081 \times 0.303 = 0.935$ volt.

$$\text{Voltage of electrolytic cell} = \frac{25.9}{100.0} \times 0.935 = 0.242 \text{ volt.}$$

Part 4. Measuring Current with a Potentiometer:

Distance from left end of scale to contact point when potentiometer is in balance = 60.4 cm. (Ave. of 3 readings)
Total voltage across slide wire (see Part 3) = 0.935 volt.

$$\text{Voltage across "B" coil} = \frac{60.4}{100.0} \times 0.935 = 0.565 \text{ volt.}$$

Resistance of "B" coil (see Part 1) = 4.592 ohms

$$\text{Current flowing through "B" coil and lamp} = \frac{E}{R} = \frac{0.565}{4.592} = 0.123 \text{ ampere}$$

Part 5. Measuring Resistance with a Potentiometer:

Since the two coils are connected in series, the same current flows through both, and hence,

$$\frac{E_B}{R_B} = \frac{E_c}{R_c} \quad \text{or} \quad R_c = \frac{E_c}{E_B} R_B$$

Voltage Across "B" Coil (E_B)

Distance from left end of scale to contact point when potentiometer is in balance = 50.95 cm. (Ave. of 3 readings)

$$\text{Voltage across coil } E_B = \frac{50.95}{100.00} \times 0.935 = 0.476 \text{ volt.}$$

Voltage Across "C" Coil (E_c)

Distance from left end of scale to contact point when potentiometer is in balance = 34.32 cm. (Ave. of 3 readings)

$$\text{Voltage across coil } E_c = \frac{34.32}{100} \times 0.935 = 0.321 \text{ volt.}$$

Resistance of "B" Coil (see Part 1) = 4.592 ohms.

$$\text{Resistance of "C" Coil} = \frac{0.321}{0.476} \times 4.592 = 3.09 \text{ ohms.}$$

This value checks quite closely with the value given for the "C" Coil at the start of the experiment, 3.080 ohms.

EXPERIMENT NO. 2

THE EFFECT OF LINE RESISTANCE ON E.M.F. INDICATING INSTRUMENTS *and* TEMPERATURE GRADIENTS IN A WIRE WOUND FURNACE

REFERENCES

Foote, Fairchild, and Harrison, *Pyrometric Practice*, National Bureau of Standards Technologic Paper No. 170, pp. 26-38, 43-46 (1921).
Wood and Cork, *Pyrometry*, pp. 41-46, 48-50 (1927).

In thermoelectric pyrometer installations, the resistance of the circuit frequently becomes of importance because of the effect it may have on the readings of certain types of e.m.f. measuring instruments. It is difficult to compensate for errors thus introduced because of the variable nature of circuit resistances. Changes in the temperatures of the conductors, and variations of the resistances of contacts, switches, and connections contribute to render the line resistance a rather uncertain factor from day to day. It is therefore important that the effect of such variations on various types of instruments be clearly understood in order that the magnitudes of the errors to be expected may be appreciated and that compensation may be applied.

Object. To study the effect of line resistance on the apparent e.m.f. of a thermocouple as indicated by several different types of instruments.

For maintaining the thermocouple junction at a fixed temperature a wire-wound, electric furnace is used. A study is made of the construction and methods of operation of this device. Determination is made of the temperature gradient curve throughout the length of the furnace.

THEORY

Line Resistance Effects. The thermocouple circuit may be considered as a resistanceless generator of an e.m.f., in series with which are the instrument and line resistances, the latter being composed of the thermocouple resistance, the resistance of the leads used to connect the thermocouple to the indicating instrument, and the resistance of any switches or connections in the circuit.

1. *Potentiometer.* In measuring the e.m.f. of a thermocouple with a potentiometer, the instrument is read when the thermocouple e.m.f. is exactly balanced by a variable and known e.m.f. impressed across the terminals of the couple. Thus, at balance, zero current is flowing in the thermocouple circuit, and hence the point of balance is theoretically uninfluenced by the resistance of the couple and its leads. From a practical viewpoint it might be added that excessive line and couple resistances decrease the sensitivity of the galvanometer to such a degree as to make it difficult to locate accurately the point of balance. However, with the normal line and couple resistances ordinarily encountered the accuracy may be regarded as uninfluenced by line and couple resistance.

2. *Millivoltmeter Calibrated to Read Voltage Across Its Terminals.* If a millivoltmeter is to be used in general laboratory work under a variety of conditions, rather than in a fixed installation, the instrument is conventionally calibrated to read voltage across its terminals. In such instances, Reading = Voltage across terminals $= \dfrac{R_g}{R_g + R_c + R_l} e.$

R_g = resistance of the instrument
R_c = resistance of the couple
R_l = resistance of the line
e = true thermocouple e.m.f.

From the above equation, it becomes apparent that the resistance of the instrument has an important bearing on the accuracy. If, for example, an instrument of 10 ohms resistance is used to measure the e.m.f. of a couple whose resistance (including line resistance) is 2 ohms, the reading will be $\left(\frac{10}{12}\right) e$. If a 500 ohm instrument is used instead, the reading will be $\left(\frac{500}{502}\right) e$, and the error will be greatly reduced.

3. *Millivoltmeter Calibrated for a Definite External Resistance.* If a millivoltmeter is to be used in a fixed thermocouple installation rather than for general laboratory use, it is customary to calibrate the scale to read true e.m.f. if the external resistance has some specified value. For example, it is common practice to calibrate millivoltmeters designed for thermocouple installations for an external resistance of 50 ohms. After the installation is made, the resistance of the couple and leads is measured, and any deficiency in resistance below the prescribed 50 ohms is made up by adding the necessary resistance in series with the instrument. For example, if the external resistance were 15 ohms, an auxiliary resistance of 50 − 15 = 35 ohms would be placed in series with the instrument, thus bringing the total external resistance up to the prescribed value of 50 ohms.

In installations of this type, the readings will exactly equal the true e.m.f. as long as the external resistance is at its prescribed value. However, should the resistance change, the readings will be in error. The magnitude of the error will depend on the change in resistance, and also upon the resistance of the meter.

$$\text{Reading} = \left[\frac{R_g + R'_0}{R_g}\right] \times \text{potential drop across terminals.}$$

R_g = resistance of the instrument.

R'_0 = external resistance for which the instrument was calibrated.

$$\text{Potential drop across terminals} = \left[\frac{R_g}{R_g + R'}\right] \times \text{true e.m.f.}$$

R' = the actual external resistance.

Combining the two above equations,

$$\text{Reading} = \left[\frac{R_g + R'_0}{R_g + R'}\right] \times \text{true e.m.f.}$$

If $R_0 = R'_0$, the reading equals the true e.m.f.

In order to demonstrate the importance of instrument resistance, the following illustration may be offered. Assume that two instruments are available, one of 500 ohms resistance, and the other of 10 ohms resistance, and assume further that both are calibrated to read correctly for an external resistance of 2 ohms. As long as the actual line and couple resistance is 2 ohms both instruments will read the true e.m.f. However, if the external resistance rises to 3 ohms, both instruments will give a low reading. The high resistance instrument will indicate $\left[\frac{500 + 2}{500 + 3}\right]$ × true e.m.f., whereas the low resistance instrument will indicate $\left[\frac{10 + 2}{10 + 3}\right]$ × true e.m.f. It is apparent that while both instruments read low, the error is much less in the case of the high resistance instrument than in the case of the low resistance instrument.

The Wire-Wound Tube Furnace. For the heating of small objects to temperatures below 1200° C., the wire-wound tube furnace is a most satisfactory and inexpensive piece of equipment. By proper current adjustments the temperature may be very accurately adjusted and held constant over long periods. An additional advantage is that any desired atmosphere may readily be maintained within the tube by the circulation of gas through it.

The furnace is constructed about a thin-walled refractory core in the form of a cylindrical tube. For the construction of this core, porcelain, bonded alumina ("Alundum"), bonded silicon carbide ("Carborundum" or "Crystolon"), or fused silica ("Vitreosil") have been successfully used. The heating element itself consists of a single-layer winding of a nickel-chromium alloy (Nichrome or Chromel) in the form of either a flat ribbon or a wire. If wire is used it is generally wound into the form of a small helix about four times its own diameter before applying to the furnace. Refractory cores may be obtained which have grooves running around their outside surfaces into which such an element may be snugly fitted. The element is then entirely covered with a layer of a high temperature refractory cement such as "Alundum." This completed core is mounted in a suitable support and surrounded by a good thermal insulator which is capable of withstanding high temperatures. Asbestos flour or diatomaceous earth (Sil-o-cel powder) are satisfactory.

In the design of such a furnace the rough rule may be followed that for a core 1″ in diameter which is to be heated to a maximum of 1100° C. provision should be made for the generation of about 100 watts of energy per inch of core length. For a tube 2″ in diameter about 150 watts per inch of length will be required. From these values the current in the element may be calculated from the voltage which is to be used. The size of wire required to carry this current may then be estimated from tables supplied by the manufacturers of the wire. Such tables list the current required to heat the wire to 1200° C., its maximum safe temperature, when horizontally stretched *in air*. When the wire is used in a tube furnace of the form described, it is safe to assume the current carrying capacity to be one-half that permissible when the wire is heated in air. After the wire size has been determined, the length required may be calculated from its resistance per foot and the current-voltage relationships in the furnace. In making these calculations, it must be remembered that the furnace design is based on operation at its maximum temperature and that the wire resistance to be used is the value at that temperature. The core is surrounded with three or four inches of thermal insulation.

Procedure. A 14 gage chromel-alumel thermocouple is inserted into the furnace tube to such a depth that the welded junction is at the center of the tube. The cold junction of the thermocouple is placed at the center of the tube passing through a large cold junction can, which is filled with ice. The ends of the tube passing through the cold junction can are plugged with cotton to prevent air circulation. The two lead wires from the thermocouple are connected to a portable potentiometer. The ends of the furnace tube are carefully plugged with asbestos in order to prevent heat loss by air infiltration. The temperature is brought to approximately 600° C. By adjusting the reactance or rheostat in series with the tube furnace, the temperature is brought to some steady value in the neighborhood of 600° C. It is not important that the temperature be exactly at 600° C., but it is important to regulate the current till a *constant* temperature is maintained. No attempt should be made to take data on the effect of variations in line resistance till constant temperature conditions have been attained. It is recommended that temperature readings be recorded at one minute intervals, and that no data on line resistance effects be taken till the time-temperature record shows a steady temperature condition. The trend of the temperature observations either upwards or downwards will indicate how the current is to be adjusted, and it will soon be possible by cut and try to adjust the current to such a value that the temperature remains steady.

The e.m.f. indications of the chromel-alumel couple are to be converted into temperature using the table included with Experiment No. 3.

Part 1. The Potentiometer. A resistance box is placed in series with the potentiometer in order that the line resistance can be increased by definite and known increments. After the furnace temperature has assumed a steady value in the vicinity of 600° C., readings of the potentiometer are made as rapidly as possible with the following successive settings of the variable resistance box: 0, 2, 5, 10, 15, 20, 30, 40, 50, 60, 80, 100 and 0 ohms. The first and last readings of the series, with a setting of 0 ohms, must be within 0.1 millivolt. If they do not agree within that limit, the temperature was not regulated to the necessary degree of constancy and a new set must be taken when conditions are more constant.

Part 2. Low Resistance Millivoltmeter Calibrated to Read Voltage at Its Terminals. The two thermocouple lead wires are connected to the two center terminals of a double pole double throw switch. The two terminals at one end of the switch are connected directly to a portable potentiometer, while the two terminals at the other end of the switch are connected to a low resistance millivoltmeter, with the variable resistance box in series. With this arrangement, it is possible to connect the thermocouple rapidly to either of the two instruments. The potentiometer serves to tell whether the temperature has the necessary degree of constancy.

The heating current should be adjusted till a steady e.m.f. reading (as given by the potentiometer) is attained, within 0.1 millivolt of the e.m.f. registered at the start of the readings taken in Part 1. The following readings are recorded in as rapid succession as possible: potentiometer reading, millivoltmeter reading with 0, 2, 5, 10, 15, 20, 30, 40, 50, 60, 80, 100 ohms in series, potentiometer reading. Here again, the two potentiometer readings constituting the start and finish of the series must agree within 0.1 millivolt. Failure to agree within this limit requires that a new series of readings be taken when the temperature has been regulated to a better degree of constancy. As part of the data, the resistance of the instrument (millivoltmeter) is to be noted.

Part 3. High Resistance Millivoltmeter Calibrated to Read Voltage at Its Terminals. The procedure to be followed is identical with that outlined for Part 2.

Part 4. High Resistance Millivoltmeter Calibrated for a Definite External Resistance. The procedure to be followed is identical with that outlined for Parts 2 and 3. The data will indicate how resistances above and below the prescribed value will affect the instrument readings.

Part 5. Temperature Gradients in the Electric Furnace. With the furnace still operating at a steady temperature, the thermocouple is connected to the potentiometer, and then pushed back until its tip touches the plug in the back end of the furnace tube. The couple is allowed to remain in this position until a steady e.m.f. is assumed. After recording the e.m.f., the couple is withdrawn an inch and allowed to remain in that position until the e.m.f. assumes a constant value. Successive readings are taken at one inch intervals until the front end of the tube is reached. Throughout this work it is important to keep the front end plugged with asbestos.

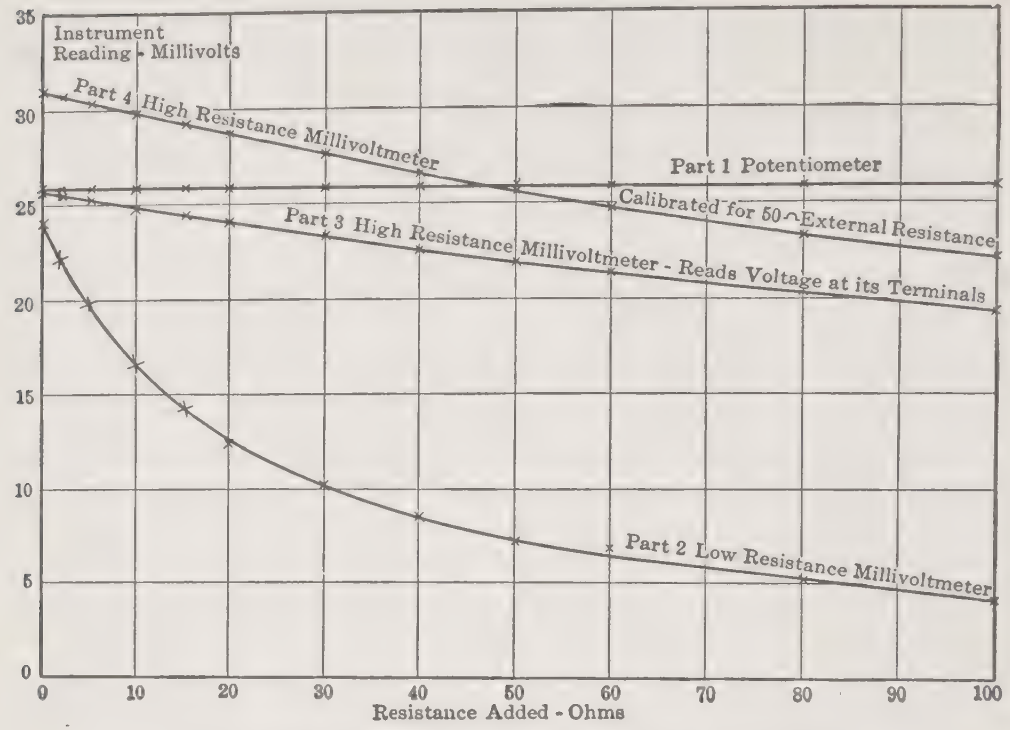

FIG. 9.—Effect of line resistance on instrument readings.

REPORT

1. The relation between instrument reading and line resistance is to be plotted for each of the instruments employed in this experiment.
2. Assuming that the potentiometer reading with 0 ohm in series represents the correct e.m.f. for each group of observations, the percentage error (plus or minus) is to be computed for each instrument when resistances of 0, 10, 50, and 100 ohms are inserted in the circuit.
3. The temperature gradient curve of the furnace is to be plotted, with temperature as ordinates and distances from the rear end of the furnace tube as abscissae.

Sample Data and Calculations

Resistance Added, Ohms	Instrument Readings			
	Part 1	Part 2	Part 3	Part 4
0	25.9 m.v.	24.2 m.v.	25.8 m.v.	31.1 m.v.
2	25.9	22.3	25.7	30.8
5	25.9	19.8	25.4	30.5
10	25.9	16.6	25.0	29.9
15	25.9	14.4	24.6	29.3
20	25.9	12.6	24.2	28.8
30	25.9	10.2	23.4	27.7
40	25.9	8.5	22.7	26.7
50	25.9	7.3	22.1	25.8
60	25.9	6.9	21.4	24.9
80	25.9	5.2	20.3	23.4
100	25.9	4.3	19.3	22.1
0	25.9	24.2	25.8	31.1
Potentiometer Reading at Start.......	25.9	25.9	26.0	25.9
Potentiometer Reading at End........	25.9	25.9	26.0	25.9

Millivoltmeter Resistances

(a) Low resistance instrument, Part 2.........................	19.8 ohms
(b) High resistance instrument, Part 3.........................	283 “
(c) High resistance instrument calibrated for 50 ohms external resistance, Part 4...................................	233 “

Calculation of Per Cent Error

$$\% \text{ Error} = \frac{\text{Observed Value} - \text{Correct Value}}{\text{Correct Value}} \times 100$$

Instrument	Per Cent Error			
	0 ohm	10 ohms	50 ohms	100 ohms
Potentiometer (Part 1).......	0.00%	0.00%	0.00%	0.00%
Low resistance millivoltmeter calibrated to read voltage at its terminals (Part 2)......	−6.6	−35.9	−71.7	−83.4
High resistance millivoltmeter calibrated to read voltage at its terminals (Part 3)......	− 0.8	− 3.8	−15.0	−25.8
High resistance millivoltmeter calibrated to read correctly with 50 ohms external resistance (Part 4)..........	+20.1	+15.4	− 0.4	−14.7

PART 5

TEMPERATURE SURVEY OF TUBE FURNACE

Distance from Rear End	Thermocouple E.M.F.	Temperature
0 inches	15.6 m.v.	381° C.
1	20.1	488
2	23.0	556
3	24.4	588
4	25.5	614
5	26.0	626
6	25.9	624
7	24.9	600
8	22.9	553
9	19.0	462
10	12.5	308

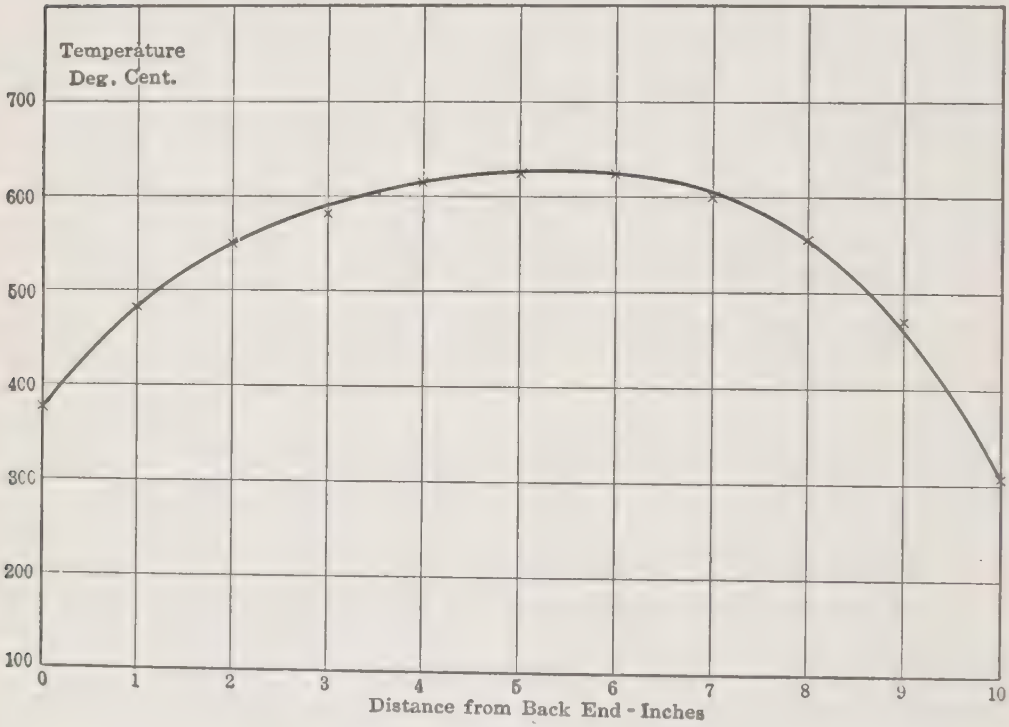

FIG. 10.—Temperature survey of tube furnace.

EXPERIMENT NO. 3

The Primary Calibration of a Thermocouple

REFERENCES

Adams, Tables and Curves for Use in Measuring Temperatures with Thermocouples, Am. Inst. Mining and Met. Engineers, Volume on Pyrometry, p. 165 (1920). This article explains the use of standard reference tables in conjunction with a deviation curve.

Roeser and Wensel, Methods of Testing Thermocouples and Thermocouple Materials, National Bureau of Standards Research Paper RP768 (1935). Also in Jour. of Res. Nat. Bur. Stnds., Vol. 14, p. 247 (1935).

Roeser and Wensel, Reference Tables for Platinum to Platinum-Rhodium Thermocouples, National Bureau of Standards Research Paper No. 530 (1933). Also in Bur. Stnds. Jour. Res., Vol. 10, p. 275 (1933).

Roeser, Dahl, and Gowens, Standard Tables for Chromel-Alumel Thermocouples, National Bureau of Standards Research Paper RP767 (1935). Also in Jour. of Res. Nat. Bur. Stnds., Vol. 14, p. 239 (1935).

Standard Conversion Tables for L. & N. Thermocouples. Issued by Leeds and Northrup Co., Philadelphia, Pa., Booklet No. 11031.

Object. The main object of this experiment is to acquaint the student with the calibration of thermocouples according to the primary method of standardization. It is, however, deemed expedient to precede a discussion of calibration methods by a brief consideration of fundamental thermoelectric principles.

THEORY

In 1821, Seebeck discovered that if a circuit is formed by individually fusing the two ends of a wire to the two ends of another wire made of different metal, and if one of the junctions thus formed is heated to a higher temperature than the other junction, an electric current will flow in the circuit. For example, if such a circuit is constructed from copper and nickel wire, it is found that the current will flow from the nickel to the copper at the hotter of the two junctions, and from copper to nickel at the cooler of the two junctions.

Two causes contribute to the production of the e.m.f. which actuates the current in such a circuit. First, an e.m.f. is developed when two different metals are brought into contact, and the magnitude of this e.m.f. is dependent upon the nature of the metals and the temperature; second, when a wire of homogeneous material is heated at one end an e.m.f. exists between the hot and cold ends of the wire the magnitude of which depends on the metal and the temperature difference between the ends. The first of these e.m.f.s is known as the Peltier e.m.f. and the second as the Thomson e.m.f.

The net e.m.f. developed in a simple thermoelectric circuit composed of two metals only is therefore the algebraic sum of the following four e.m.f.s: (1) The Peltier e.m.f. at the hot junction. (2) The Peltier e.m.f. at the cold junction. (3) The Thomson e.m.f. in one wire. (4) The Thomson e.m.f. in the other wire.

The magnitude of the net e.m.f., which is the algebraic sum of the above enumerated four individual e.m.f.s, is determined by the following three factors: (1) The temperature of the hot junction. (2) The temperature of the cold junction. (3) The nature of the two wires comprising the thermocouple.

If the temperature of one junction is kept constant, say at the melting point of ice (0° C.), the temperature of the other junction can be determined by measuring the e.m.f. developed in the circuit. In other words, when the temperature of the cold junction of a thermocouple is kept constant, the temperature of the hot junction is some function of the e.m.f. developed. This is the basic principle of thermoelectric pyrometry.

It perhaps should be emphasized that the temperature assumed by the thermoelements at various points between the hot and cold junctions is without effect on the e.m.f. developed; it is the *junction* temperatures rather than the temperature of intermediate points between the hot and cold junctions that will determine the magnitude of the e.m.f. set up in a given thermocouple. As a corollary, it is evident that the requirement of cold junction control in utilizing a thermocouple will not be fulfilled by maintaining a constant temperature at some point in the thermoelements between the hot and cold junctions; the cold junction itself must be held at a steady, known temperature (usually 0° C. in laboratory work).

Thermoelectric Power. The thermoelectric power of a given thermocouple at any specific temperature is the slope of the e.m.f.-temperature curve (e.m.f.s as ordinates and temperatures as abscissae). In other words, the thermoelectric power of a couple at some specific temperature is the increment in e.m.f. produced by a 1 deg. Centigrade rise in temperature of the hot junction.

If the equation relating e.m.f. and temperature is known, the thermoelectric power may be determined by differentiating the equation. Conversely, if the equation relating thermoelectric power and temperature is known, the relation between e.m.f. and temperature may be determined by simple integration.

Neutral Point. In certain thermoelectric combinations the e.m.f. produced over ordinary temperature ranges may first increase with the temperature, gradually approach a maximum, and then decrease with a further increase of temperature. On further increasing the temperature the e.m.f. may fall to zero and then increase in the negative direction. The temperature at which the rate of increase of the e.m.f. with respect to temperature equals zero ($dE/dt = 0$) is called the neutral point. An example of such a thermoelectric combination is the iron-copper thermocouple. Any couple exhibiting such a characteristic within the range of temperatures to be measured cannot be used as a temperature measuring device, since two temperatures correspond to a given e.m.f. value.

The Effect of a Third Metal in the Circuit. The discussion thus far has been confined to an extremely simple type of circuit composed of two wires only, and with the two wires making direct contact with one another at both hot and cold junctions. When it is desired to measure thermoelectric e.m.f.s, it becomes necessary to introduce an e.m.f. measuring device into the circuit, and this is customarily done by connecting a copper lead wire to the cold free end of each of the thermoelements and then running the two copper leads to the measuring instrument. Thus, copper, a third metal is introduced into the circuit, and we no longer have a single direct contact between the two thermoelements at the cold end, but instead, two junctions, one between each thermoelement and its attached copper lead wire. With this type of circuit, the effective cold junction of the system will be at the union between the copper leads and the thermoelements, not at the instrument terminals. Furthermore, the introduction of a third metal (copper in the present illustration) into the thermoelectric circuit is without effect on the e.m.f. produced provided that the two points where it is joined to the other metals comprising the circuit have the same temperature.

Cold Junction Correction. Calibration curves and tables for thermocouples are usually based on a cold junction temperature of 0° C. If the couple subsequently is utilized with the cold junction at some temperature other than 0° C., it becomes necessary to apply a cold junction correction. This is accomplished by adding to the observed e.m.f., the e.m.f. corresponding to the cold junction temperature, as read from the curve or table. The summation represents the e.m.f. the couple would generate if the temperature were at 0° C., and this value may then be used to evaluate the true hot junction temperature, by reference to the curve or table.

$$E_{(t,\,0)} = E_{(t,\,t')} + E_{(t',\,0)}$$

$E_{(t,\,0)}$ = e.m.f. generated with the hot junction at temperature t and the cold junction at 0° C.

$E_{(t,\,t')}$ = e.m.f. generated with the hot junction at temperature t and the cold junction at t'.

$E_{(t',\,0)}$ = e.m.f. generated with the hot junction at temperature t' and the cold junction at 0° C.

It should be insisted that it is incorrect in principle to add the cold junction temperature to the apparent or indicated temperature, the latter being obtained by utilizing the observed e.m.f. with the calibration curve or table. Such a direct addition of temperatures would be correct in principle if the temperature-e.m.f. relation were exactly linear. However, no thermocouple combination exhibits an exactly linear relationship. Of the various thermocouple combinations of industrial importance, the chromel-alumel couple approaches most closely a linear temperature-e.m.f. relation. In using this particular couple in industrial work where inaccuracies of 2 to 3 deg. C. are permissible, cold junction corrections are sometimes applied by a direct addition of cold junction temperature to indicated temperature.

The Two General Methods of Calibrating Thermocouples. (1) *Primary Standardization.* The primary calibration of a thermocouple involves the determination of its e.m.f. at the melting or boiling points of several substances whose melting or boiling points are accurately known. Fixed points for the calibration are chosen which cover the temperature range in which the thermocouple is to be used.

It is very essential that pure materials be employed in the calibration, as the freezing and boiling points are markedly affected by the presence of impurities. The National Bureau of Standards furnishes a series of metals of certified melting point at small cost. Accurate calibration work makes it highly advisable to utilize metals from this source.

(2) *Secondary Standardization.* If an accurately calibrated thermocouple is available, it may be utilized to calibrate other thermocouples by a direct comparison method. The hot junction of the standard and the couple to be calibrated are tied together, and placed at the center of an electrically heated tube furnace whose temperature may be accurately controlled. At various suitably spaced steady temperatures, simultaneous e.m.f. readings are taken on the two thermocouples.

Reduction of Primary Standardization Data to Usable Form. In handling the data obtained in a primary standardization, it is not customary to make a direct plot of the experimentally determined e.m.f.s versus the corresponding temperatures. Such a procedure would be objectionable because a rather large number of fixed points would have to be employed in order that the curve could be drawn with a satisfactory degree of precision. The conventional procedure is to utilize a standard temperature-e.m.f. table in conjunction with a deviation curve that is based on the experimental data. This method of procedure is outlined more fully below.

Accompanying this experiment are standard reference tables for the platinum versus 90% platinum + 10% rhodium thermocouple, the chromel- alumel thermocouple, the iron-constantan thermocouple, and the copper-constantan thermocouple. These reference tables are based on average values obtained from many thermocouples. Any given thermocouple will deviate somewhat from the temperature-e.m.f. relationship indicated by these tables. It is therefore evident that in precise work the tables cannot be used directly for any particular couple. However, if the actual e.m.f. of the couple at several fixed freezing or boiling points is determined experimentally, it is possible to plot a curve for the specific couple under consideration, showing the difference between the standard table and the couple, as a function of the couple e.m.f. When such a curve has been plotted, it is a simple matter to determine the temperature corresponding to any particular e.m.f. In more detail, the method is as follows:

(1) Determine by direct experiment the e.m.f. of the couple at a number of fixed points covering the range of temperatures in which the couple is to be used. In many calibrations, the freezing points of zinc, antimony, and copper are suitable. Aluminum may be substituted for antimony if desired.

(2) From the calibration data, compute the difference between the standard reference table and the indication of the thermocouple.

$$\Delta E = (\text{e.m.f. indicated by reference table}) - (\text{e.m.f. of the couple being calibrated}) = E_r - E_x.$$

If data were obtained at the freezing points of zinc, antimony, and copper,

$$(\Delta E)_{Zn} = (E_r)_{Zn} - (E_x)_{Zn}$$

$$(\Delta E)_{Sb} = (E_r)_{Sb} - (E_x)_{Sb}$$

$$(\Delta E)_{Cu} = (E_r)_{Cu} - (E_x)_{Cu}$$

$(\Delta E)_0$, the deviation at the ice point, equals zero.

(3) Plot a difference, or deviation curve for the particular couple under consideration, with values of ΔE as ordinates, versus values of E_x as abscissae.

(4) Suppose now that it is desired to determine the temperature corresponding to some specific thermocouple e.m.f. $(E_x)_1$. Refer to the deviation curve and read off $(\Delta E)_1$. Compute the e.m.f. the standard reference couple would generate from the equation

$$(E_r)_1 = (E_x)_1 + (\Delta E)_1$$

Then using this computed value $(E_r)_1$, look up the corresponding temperature in the standard reference table.

Method of Determining E.M.F.s at Melting or Freezing Points. Pure samples of metals are used as fixed temperature points in the calibration of a thermocouple. The calibration of the couple at the standard tem-

E.M.F. VALUES AT FIXED POINTS AS TAKEN FROM STANDARD TABLES

Cold Junction Temperature = 0° C.

Freezing Point	Temperature		Platinum — 90% Platinum + 10% Rhodium	Chromel-Alumel	Iron-Constantan	Copper-Constantan
	Deg. C.	Deg. F.				
Tin	231.85	449.33	1.708	9.41	12.54	11.01
Cadmium	320.9	609.6	2.506	13.08	17.45	16.08
Lead	327.3	621.1	2.566	13.33	17.81	16.46
Zinc	419.45	787.01	3.436	17.22	22.89	
Antimony	630.5	1166.9	5.535	26.20	34.97	
Aluminum	660.15	1220.27	5.842	27.46	36.75	
Copper	1083	1981	10.535	44.50	63.43	

peratures is accomplished by determining the e.m.f. developed by the couple when its hot junction is at the temperature of the melting point of the metal under reducing conditions and its cold junction is at 0° C.

The melting point is detected by heating or cooling the sample (which is covered with powdered graphite) at a uniform rate through the temperature range in which the transition takes place. At the melting or freezing point the uniform temperature change will be arrested due to the absorption or evolution of the heat of fusion. A heating or cooling curve in which time is plotted against the corresponding thermocouple e.m.f. will show a change in slope at the transition point. The e.m.f. corresponding to the known melting temperature is indicated by the straight, flat portion of the curve beyond the point of change in the direction of curvature.

Procedure. In this experiment, the student will be assigned a thermocouple which is to be calibrated according to the primary method of standardization. The calibration points used will be the freezing points of zinc (419.5° C.), antimony (630.5° C.) and copper (1083.0° C.). Aluminum (660.2° C.) may be substituted for the antimony.

Each metal is contained in a crucible of Acheson graphite. Suitable dimensions for the crucibles are given in Reference No. 2, p. 257. The surface of the metal is covered with a layer of powdered graphite to prevent oxidation of the metal. Each crucible is provided with a tightly fitting cover, also turned out of Acheson graphite. Through the crucible cover, and extending within ¼ inch of the bottom of the crucible on the inside, is a graphite protection tube. The crucibles containing the zinc, antimony, and copper standards are placed in individual electric furnaces. To prevent rapid external oxidation of the graphite crucibles, they are set on graphite pedestals, and also covered with graphite blocks, drilled to permit entry of the thermocouple.

The thermocouple to be calibrated is provided with a primary protection tube. At the zinc and antimony points, fused silica tubes are satisfactory, while at the copper point, glazed alundum is to be preferred over fused silica, as the latter becomes porous to gases at high temperatures. The thermocouple, in its refractory protection tube, is inserted into the graphite tube of the crucible containing the zinc standard.

The cold junction (the joints between the copper lead wires and the thermoelements) is cooled to 0° C. by means of a suitable cold junction jar. The thermocouple is then connected to a portable potentiometer, which is used throughout the experiment to measure the e.m.f. of the thermocouple.

The heating current is turned on in the electric furnace containing the zinc standard, and adjusted to a value suggested by the instructor. Observations of thermocouple e.m.f. are taken every minute as the anticipated fusion point is approached. Fusion will be indicated by constancy of e.m.f. over a period of several minutes. After the sample is completely melted, as indicated by a rising e.m.f., the heating current is shut off and the sample allowed to cool. Observations are taken on cooling, and freezing is indicated by constancy of e.m.f. over a period of several minutes. If experimental conditions are correct, the e.m.f. during fusion should be very nearly the same as the e.m.f. during freezing. For example, when working with chromel-alumel couples, and using a potentiometer that can be read to 0.1 m.v., the two values should not differ by more than 0.1 m.v. Should the two values not agree, that obtained on cooling is to be preferred since conditions are more uniform on cooling than on heating.

The procedure is substantially the same for the antimony and copper points. For the copper point, the furnace described in Reference 2, pages 258 and 259, has been very satisfactory.

REPORT

1. Plot the heating and cooling curves of the metals used, using e.m.f.s as ordinates and time as abscissae. Designate the freezing point by means of an arrow, and affix thereto the corresponding e.m.f.
2. Fill in the following tabulation

Type of Couple____________ Couple No.____________

Fixed Point	E.M.F. of Couple Calibrated, E_x	E.M.F. of Standard Reference Couple, E_r	$\Delta E = E_r - E_x$
Ice.	0	0	0
Zinc.			
Antimony.			
Aluminum.			
Copper.			

3. Utilizing the values in the above table, plot the deviation curve for the couple, ΔE on the vertical axis, and E_x on the horizontal axis. *Connect the plotted points with straight lines.*

4. Illustrate by means of a specific example how an e.m.f. reading is to be converted into temperature.
5. When measuring a temperature with the high range of the portable potentiometer, the sensitivity of the instrument is such that e.m.f.

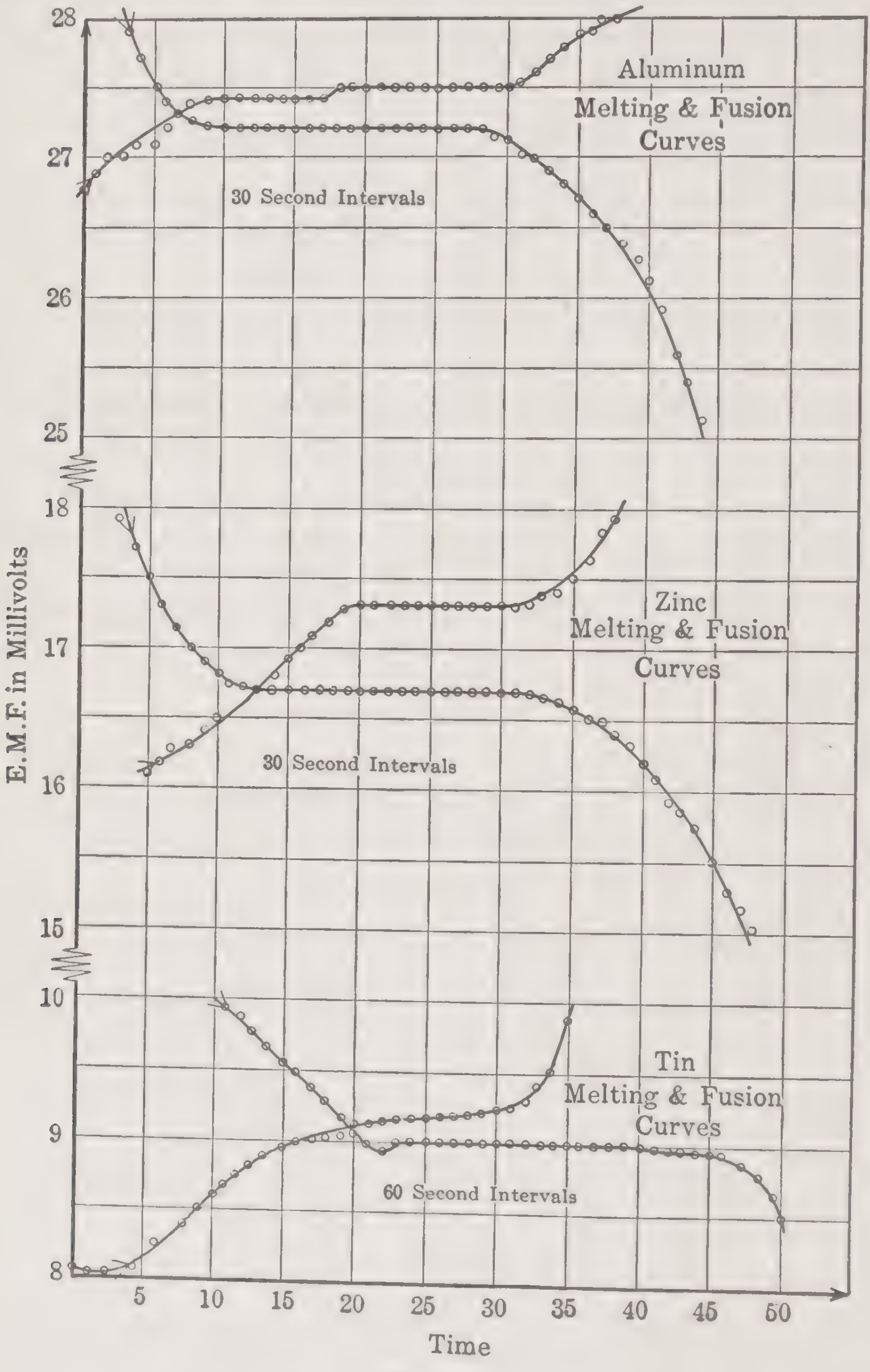

FIG. 11

readings may be duplicated under constant conditions with ± 0.1 m.v. On the basis of a probable error of ± 0.1 m.v. in the e.m.f. reading of a chromel-alumel couple, what is the probable error of the temperature being measured?

6. When using the low range of the portable potentiometer, e.m.f. readings under constant conditions may be duplicated within ± 0.01 m.v. What is the probable error of a temperature measurement based on the low range?

SAMPLE DATA AND CALCULATIONS

On the curve sheet on page 36 are given heating and cooling curves for the thermocouple calibrated in the zinc, tin, and aluminum standards.

Type of Couple: Chromel-Alumel Couple No. 5

Fixed Point	E.M.F. of Couple Calibrated E_x	E.M.F. of Standard Reference Couple E_r	$\Delta E = E_r - E_x$
Ice................	0 m.v.	0 m.v.	0 m.v.
Zinc................	17.2	17.2	0
Antimony...........	26.0	26.2	+0.2
Copper..............	44.4	44.5	+0.1

The deviation or difference curve for the couple is now plotted, with values of ΔE as ordinates and values of E_x as abscissae.

Method of Converting E.M.F. into Temperature: If the couple calibrated indicates an e.m.f. of 35.4 m.v., what is the hot junction temperature? Referring to the deviation curve, we see that $\Delta E = +.15$ m.v.

$$E_r = E_x + \Delta E = 35.4 + 0.15 = 35.55 \text{ m.v.}$$

Referring to the standard table for chromel-alumel couples we see that the temperature corresponding to 35.55 m.v. is 855° C. (Table 2.)

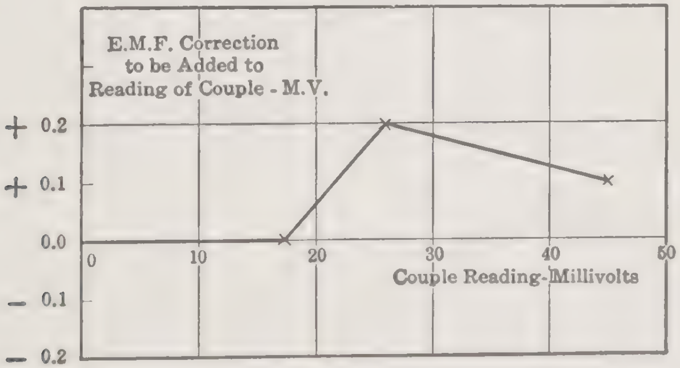

FIG. 12

TABLE 1—PLATINUM VS. 90% PLATINUM + 10% RHODIUM THERMOCOUPLES *

E.m.f.s are expressed in microvolts (1 millivolt = 1000 microvolts).
Temperatures are in Deg. Cent.
Cold junction temperature is 0 Deg. Cent.

[Emfs are expressed in microvolts and temperatures in °C. Cold junctions at 0° C.]

E Micro-volts	0	1,000	2,000	3,000	4,000	5,000	6,000	7,000	8,000	E Micro-volts
0	0	146. 9	265. 0	373. 7	477. 7	578. 1	675. 3	769. 5	861. 0	0
	17. 7	*12. 5*	*11. 2*	*10. 5*	*10. 2*	*9. 9*	*9. 5*	*9. 3*	*9. 0*	
100	17. 7	159. 4	276. 2	384. 2	487. 9	588. 0	684. 8	778. 8	870. 0	100
	16. 7	*12. 3*	*11. 1*	*10. 5*	*10. 2*	*9. 8*	*9. 5*	*9. 2*	*9. 0*	
200	34. 4	171. 7	287. 3	394. 7	498. 1	597. 8	694. 3	788. 0	879. 0	200
	15. 8	*12. 1*	*11. 0*	*10. 5*	*10. 1*	*9. 8*	*9. 5*	*9. 2*	*9. 0*	
300	50. 2	183. 8	298. 3	405. 2	508. 2	607. 6	703. 8	797. 2	888. 0	300
	15. 2	*12. 0*	*11. 0*	*10. 5*	*10. 1*	*9. 8*	*9. 5*	*9. 2*	*9. 0*	
400	65. 4	195. 8	309. 3	415. 7	518. 3	617. 4	713. 3	806. 4	897. 0	400
	14. 6	*11. 8*	*10. 9*	*10. 4*	*10. 1*	*9. 7*	*9. 4*	*9. 2*	*8. 9*	
500	80. 0	207. 6	320. 2	426. 1	528. 4	627. 1	722. 7	815. 6	905. 9	500
	14. 1	*11. 7*	*10. 8*	*10. 4*	*10. 0*	*9. 7*	*9. 4*	*9. 1*	*8. 9*	
600	94. 1	219. 3	331. 0	436. 5	538. 4	636. 8	732. 1	824. 7	914. 8	600
	13. 7	*11. 6*	*10. 7*	*10. 3*	*10. 0*	*9. 7*	*9. 4*	*9. 1*	*8. 9*	
700	107. 8	230. 9	341. 7	446. 8	548. 4	646. 5	741. 5	833. 8	923. 7	700
	13. 3	*11. 5*	*10. 7*	*10. 3*	*9. 9*	*9. 6*	*9. 4*	*9. 1*	*8. 9*	
800	121. 1	242. 4	352. 4	457. 1	558. 3	656. 1	750. 9	842. 9	932. 6	800
	13. 0	*11. 3*	*10. 7*	*10. 3*	*9. 9*	*9. 6*	*9. 3*	*9. 1*	*8. 8*	
900	134. 1	253. 7	363. 1	467. 4	568. 2	665. 7	760. 2	852. 0	941. 4	900
	12. 8	*11. 3*	*10. 6*	*10. 3*	*9. 9*	*9. 6*	*9. 3*	*9. 0*	*8. 8*	
1,000	146. 9	265. 0	373. 7	477. 7	578. 1	675. 3	769. 5	861. 0	950. 2	1,000

* From Reference Tables for Platinum to Platinum-Rhodium Thermocouples, by Roeser and Wensel, *Bureau of Standards Journal of Research*, Vol. 10, p. 275 (1933).

TABLE 1 (*Continued*)

E Micro-volts	9,000		10,000		11,000		12,000		13,000		14,000		15,000		16,000		17,000		E Micro-volts
0	950.2		1,037.2		1,122.3		1,206.4		1,290.0		1,373.8		1,458.0		1,542.6		1,627.8		0
		8.8		*8.6*		*8.5*		*8.3*		*8.3*		*8.4*		*8.4*		*8.5*		*8.6*	
100	959.0		1,045.8		1,130.8		1,214.7		1,298.3		1,382.2		1,466.4		1,551.1		1,636.4		100
		8.8		*8.6*		*8.4*		*8.4*		*8.4*		*8.4*		*8.4*		*8.5*		*8.5*	
200	967.8		1,054.4		1,139.2		1,223.1		1,306.7		1,390.6		1,474.8		1,559.6		1,644.9		200
		8.7		*8.5*		*8.4*		*8.3*		*8.4*		*8.4*		*8.5*		*8.5*		*8.6*	
300	976.5		1,062.9		1,147.6		1,231.4		1,315.1		1,399.0		1,483.3		1,568.1		1,653.5		300
		8.8		*8.6*		*8.4*		*8.4*		*8.4*		*8.4*		*8.5*		*8.5*		*8.6*	
400	985.3		1,071.5		1,156.0		1,239.8		1,323.5		1,407.4		1,491.8		1,576.6		1,662.1		400
		8.7		*8.5*		*8.4*		*8.4*		*8.3*		*8.4*		*8.4*		*8.5*		*8.6*	
500	994.0		1,080.0		1,164.4		1,248.2		1,331.8		1,415.8		1,500.2		1,585.1		1,670.7		500
		8.7		*8.5*		*8.4*		*8.3*		*8.4*		*8.4*		*8.5*		*8.6*		*8.6*	
600	1,002.7		1,088.5		1,172.8		1,256.5		1,340.2		1,424.2		1,508.7		1,593.7		1,679.3		600
		8.6		*8.5*		*8.4*		*8.4*		*8.4*		*8.5*		*8.5*		*8.5*		*8.6*	
700	1,011.3		1,097.0		1,181.2		1,264.9		1,348.6		1,432.7		1,517.2		1,602.2		1,687.9		700
		8.7		*8.4*		*8.4*		*8.3*		*8.4*		*8.4*		*8.4*		*8.5*		*8.6*	
800	1,020.0		1,105.4		1,189.6		1,273.2		1,357.0		1,441.1		1,525.6		1,610.7		1,696.5		800
		8.6		*8.5*		*8.4*		*8.4*		*8.4*		*8.4*		*8.5*		*8.6*		*8.6*	
900	1,028.6		1,113.9		1,198.0		1,281.6		1,365.4		1,449.5		1,534.1		1,619.3		1,705.1		900
		8.6		*8.4*		*8.4*		*8.4*		*8.4*		*8.5*		*8.5*		*8.5*		*8.6*	
1,000	1,037.2		1,122.3		1,206.4		1,290.0		1,373.8		1,458.0		1,542.6		1,627.8		1,713.7		1,000

* From Reference Tables for Platinum to Platinum-Rhodium Thermocouples, by Roeser and Wensel, *Bureau of Standards Journal of Research,* Vol. 10, p. 275 (1933).

TABLE 2—Chromel-Alumel Thermocouples*

E.m.f.s are expressed in millivolts.
Temperatures are in Deg. Cent.
Cold junction temperature is 0 Deg. Cent.

CHROMEL VS. ALUMEL THERMOCOUPLE

Degrees, Centigrade Reference Junction 0° C.

Deg. C.	0°	100°	200°	300°	400°	500°	600°
	Millivolts.						
0°	0	4.10	8.13	12.21	16.39	20.64	24.90
2°	.08	4.18	8.21	12.29	16.47	20.73	24.99
4°	.16	4.26	8.29	12.37	16.55	20.81	25.07
6°	.24	4.34	8.37	12.45	16.64	20.90	25.16
8°	.32	4.42	8.45	12.53	16.73	20.98	25.24
10°	.40	4.51	8.53	12.62	16.82	21.07	25.33
12°	.48	4.59	8.61	12.70	16.90	21.15	25.41
14°	.56	4.67	8.69	12.78	16.99	21.24	25.50
16°	.64	4.75	8.77	12.86	17.07	21.32	25.58
18°	.72	4.83	8.85	12.95	17.16	21.41	25.67
20°	.80	4.92	8.93	13.04	17.24	21.49	25.75
22°	.88	5.00	9.01	13.12	17.32	21.58	25.84
24°	.96	5.08	9.09	13.20	17.41	21.66	25.93
26°	1.04	5.16	9.17	13.28	17.49	21.75	26.01
28°	1.12	5.24	9.25	13.36	17.58	21.83	26.09
30°	1.20	5.33	9.34	13.45	17.66	21.92	26.18
32°	1.28	5.41	9.42	13.53	17.74	22.00	26.26
34°	1.36	5.49	9.50	13.61	17.83	22.09	26.35
36°	1.44	5.57	9.58	13.69	17.91	22.17	26.43
38°	1.52	5.65	9.66	13.78	18.00	22.26	26.52
40°	1.61	5.73	9.74	13.87	18.08	22.34	26.60
42°	1.69	5.81	9.82	13.95	18.16	22.43	26.69
44°	1.77	5.89	9.90	14.03	18.25	22.52	26.77
46°	1.85	5.97	9.98	14.11	18.33	22.60	26.86
48°	1.93	6.05	10.06	14.20	18.42	22.68	26.94
50°	2.02	6.13	10.15	14.29	18.50	22.77	27.03
52°	2.10	6.21	10.23	14.37	18.58	22.86	27.12
54°	2.18	6.29	10.31	14.45	18.66	22.94	27.20
56°	2.26	6.37	10.39	14.53	18.75	23.03	27.28
58°	2.34	6.45	10.47	14.62	18.84	23.11	27.37
60°	2.43	6.53	10.56	14.71	18.93	23.20	27.45
62°	2.51	6.61	10.64	14.79	19.02	23.28	27.53
64°	2.59	6.69	10.72	14.88	19.11	23.37	27.62
66°	2.67	6.77	10.80	14.96	19.20	23.45	27.70
68°	2.76	6.85	10.88	15.05	19.28	23.54	27.79
70°	2.85	6.93	10.97	15.13	19.36	23.62	27.87
72°	2.93	7.01	11.05	15.21	19.44	23.71	27.95
74°	3.01	7.09	11.13	15.30	19.53	23.79	28.04
76°	3.09	7.17	11.21	15.38	19.61	23.88	28.12
78°	3.17	7.25	11.29	15.47	19.70	23.96	28.21
80°	3.26	7.33	11.38	15.55	19.78	24.05	28.29
82°	3.34	7.41	11.46	15.63	19.87	24.14	28.38
84°	3.42	7.49	11.54	15.72	19.95	24.22	28.46
86°	3.50	7.57	11.62	15.80	20.04	24.31	28.55
88°	3.59	7.65	11.71	15.89	20.12	24.39	28.63
90°	3.68	7.73	11.80	15.97	20.21	24.48	28.72
92°	3.76	7.81	11.88	16.05	20.30	24.56	28.80
94°	3.84	7.89	11.96	16.14	20.38	24.65	28.89
96°	3.92	7.97	12.04	16.22	20.47	24.73	28.97
98°	4.01	8.05	12.12	16.31	20.55	24.82	29.06
100°	4.10	8.13	12.21	16.39	20.64	24.90	29.14
M. V. per ° C.	**.041**	**.0403**	**.0408**	**.0418**	**.0425**	**.0426**	**.0424**

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* Reproduced from the literature of the Leeds and Northrup Co., and is based on the paper of Roeser, Dahl, and Gowens, Standard Tables for Chromel-Alumel Thermocouples, *Journal of Research, National Bureau of Standards*, Vol. 14. p. 239 (1935).

TABLE 2 (*Continued*)

CHROMEL VS. ALUMEL THERMOCOUPLE

Degrees, Centigrade Reference Junction 0° C.

Deg. C.	700°	800°	900°	1000°	1100°	1200°	1300°
	Millivolts.						
0°	29.14	33.31	37.36	41.31	45.14	48.85	52.41
2°	29.22	33.39	37.44	41.39	45.22	48.93	52.48
4°	29.31	33.47	37.52	41.47	45.29	49.00	52.55
6°	29.39	33.55	37.60	41.55	45.37	49.07	52.62
8°	29.48	33.63	37.68	41.63	45.44	49.14	52.69
10°	29.56	33.71	37.76	41.70	45.52	49.21	52.75
12°	29.64	33.79	37.84	41.78	45.59	49.29	52.82
14°	29.73	33.87	37.92	41.86	45.67	49.36	52.89
16°	29.81	33.95	38.00	41.94	45.74	49.43	52.96
18°	29.90	34.03	38.08	42.01	45.82	49.50	53.03
20°	29.98	34.12	38.16	42.08	45.89	49.57	53.10
22°	30.06	34.20	38.24	42.16	45.97	49.65	53.17
24°	30.15	34.28	38.32	42.24	46.04	49.73	53.24
26°	30.23	34.36	38.40	42.32	46.12	49.80	53.31
28°	30.32	34.44	38.48	42.40	46.19	49.87	53.38
30°	30.40	34.53	38.56	42.47	46.27	49.94	53.45
32°	30.48	34.61	38.64	42.55	46.35	50.01	53.52
34°	30.57	34.69	38.72	42.62	46.43	50.08	53.59
36°	30.65	34.77	38.80	42.70	46.50	50.15	53.66
38°	30.74	34.85	38.88	42.78	46.57	50.22	53.73
40°	30.82	34.94	38.96	42.86	46.64	50.29	53.79
42°	30.90	35.02	39.04	42.94	46.71	50.37	53.86
44°	30.98	35.10	39.12	43.02	46.79	50.44	53.93
46°	31.06	35.18	39.20	43.10	46.86	50.51	54.00
48°	31.14	35.26	39.28	43.17	46.94	50.58	54.07
50°	31.23	35.35	39.35	43.24	47.01	50.65	54.13
52°	31.31	35.43	39.43	43.32	47.08	50.72	54.20
54°	31.40	35.51	39.51	43.39	47.16	50.79	54.27
56°	31.48	35.59	39.59	43.47	47.23	50.86	54.34
58°	31.57	35.67	39.67	43.54	47.31	50.93	54.41
60°	31.65	35.75	39.75	43.62	47.38	51.00	54.47
62°	31.73	35.83	39.83	43.70	47.45	51.08	54.54
64°	31.82	35.91	39.91	43.77	47.53	51.15	54.61
66°	31.90	35.99	39.99	43.85	47.60	51.22	54.68
68°	31.99	36.07	40.07	43.92	47.68	51.29	54.75
70°	32.07	36.16	40.14	44.00	47.75	51.36	54.81
72°	32.15	36.24	40.22	44.08	47.82	51.43	54.88
74°	32.23	36.32	40.30	44.15	47.90	51.50	54.95
76°	32.31	36.40	40.38	44.23	47.97	51.57	55.02
78°	32.39	36.48	40.46	44.30	48.05	51.64	55.09
80°	32.48	36.56	40.53	44.38	48.12	51.71	55.15
82°	32.56	36.64	40.61	44.46	48.20	51.78	55.22
84°	32.65	36.72	40.69	44.53	48.27	51.85	55.29
86°	32.73	36.80	40.77	44.61	48.34	51.92	55.36
88°	32.82	36.88	40.85	44.68	48.41	51.99	55.42
90°	32.90	36.96	40.92	44.76	48.48	52.06	55.48
92°	32.98	37.04	41.00	44.84	48.56	52.13	55.55
94°	33.06	37.12	41.08	44.91	48.64	52.20	55.62
96°	33.14	37.20	41.16	44.99	48.71	52.27	55.69
98°	33.22	37.28	41.24	45.06	48.78	52.34	55.75
100°	33.31	37.36	41.31	45.14	48.85	52.41	55.81
M. V. per ° C.	.0417	.0405	.0395	.0383	.0371	.0356	.0340

TABLE 2 (Second Part)

TABLE 3—IRON-CONSTANTAN THERMOCOUPLES*

E.m.f.s are expressed in millivolts.
Temperatures are in Deg. Cent.
Cold junction temperature is 0 Deg. Cent.

IRON VS. CONSTANTAN THERMOCOUPLE

Degrees, Centigrade **Reference Junction 0° C.**

Deg. C.	0°	100°	200°	300°	400°	500°
	Millivolts.					
0°	.0	5.28	10.78	16.30	21.82	27.39
2°	.10	5.39	10.89	16.41	21.93	27.50
4°	.21	5.50	11.00	16.52	22.04	27.62
6°	.31	5.61	11.11	16.63	22.15	27.73
8°	.42	5.72	11.22	16.74	22.26	27.84
10°	.52	5.83	11.33	16.85	22.37	27.96
12°	.62	5.94	11.44	16.96	22.48	28.08
14°	.73	6.05	11.55	17.07	22.59	28.19
16°	.83	6.16	11.66	17.18	22.70	28.31
18°	.94	6.27	11.77	17.29	22.81	28.42
20°	1.04	6.38	11.88	17.40	22.92	28.53
22°	1.14	6.49	11.99	17.51	23.03	28.65
24°	1.25	6.60	12.10	17.62	23.14	28.76
26°	1.35	6.70	12.22	17.74	23.26	28.88
28°	1.46	6.81	12.33	17.85	23.37	28.99
30°	1.56	6.92	12.44	17.96	23.48	29.11
32°	1.66	7.03	12.55	18.07	23.59	29.22
34°	1.77	7.14	12.66	18.18	23.70	29.33
36°	1.87	7.25	12.77	18.29	23.81	29.45
38°	1.98	7.36	12.88	18.40	23.92	29.56
40°	2.08	7.47	12.99	18.51	24.03	29.68
42°	2.18	7.58	13.10	18.62	24.14	29.79
44°	2.29	7.69	13.21	18.73	24.25	29.91
46°	2.39	7.80	13.32	18.84	24.36	30.02
48°	2.50	7.91	13.43	18.95	24.47	30.14
50°	2.60	8.02	13.54	19.06	24.58	30.25
52°	2.71	8.13	13.65	19.17	24.69	30.37
54°	2.81	8.24	13.76	19.28	24.80	30.48
56°	2.92	8.35	13.87	19.39	24.92	30.60
58°	3.03	8.46	13.98	19.50	25.03	30.71
60°	3.14	8.57	14.09	19.61	25.14	30.83
62°	3.24	8.68	14.20	19.72	25.25	30.95
64°	3.35	8.79	14.31	19.83	25.37	31.06
66°	3.46	8.90	14.42	19.94	25.48	31.18
68°	3.56	9.01	14.53	20.05	25.59	31.30
70°	3.67	9.12	14.64	20.16	25.70	31.41
72°	3.78	9.23	14.75	20.27	25.82	31.53
74°	3.89	9.34	14.86	20.38	25.93	31.65
76°	3.99	9.45	14.98	20.49	26.04	31.76
78°	4.10	9.57	15.09	20.61	26.15	31.88
80°	4.21	9.68	15.20	20.72	26.27	32.00
82°	4.31	9.79	15.31	20.83	26.38	32.11
84°	4.42	9.90	15.42	20.94	26.49	32.23
86°	4.53	10.01	15.53	21.05	26.60	32.35
88°	4.64	10.12	15.64	21.16	26.72	32.46
90°	4.74	10.23	15.75	21.27	26.83	32.58
92°	4.85	10.34	15.86	21.38	26.94	32.69
94°	4.96	10.45	15.97	21.49	27.05	32.81
96°	5.06	10.56	16.08	21.60	27.17	32.93
98°	5.17	10.67	16.19	21.71	27.28	33.04
100°	5.28	10.78	16.30	21.82	27.39	33.16
M. V. per ° C.	.0528	.055	.0552	.0552	.0557	.0577

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* Reproduced from the literature of the Leeds and Northrup Co.

TABLE 3 (*Continued*)

IRON VS. CONSTANTAN THERMOCOUPLE

Degrees, Centigrade — Reference Junction 0° C.

Deg. C.	600°	700°	800°	900°	1000°
	Millivolts.				
0°	33.16	39.19	45.48	51.82	58.16
2°	33.28	39.32	45.61	51.95	58.29
4°	33.40	39.44	45.73	52.07	58.41
6°	33.52	39.57	45.86	52.20	58.54
8°	33.64	39.69	45.99	52.33	58.67
10°	33.75	39.82	46.11	52.45	58.79
12°	33.87	39.94	46.24	52.58	58.92
14°	33.99	40.07	46.37	52.71	59.05
16°	34.11	40.19	46.49	52.83	59.17
18°	34.23	40.32	46.62	52.96	59.30
20°	34.35	40.44	46.75	53.09	59.43
22°	34.47	40.57	46.87	53.21	59.55
24°	34.59	40.69	47.00	53.34	59.68
26°	34.70	40.82	47.13	53.47	59.81
28°	34.82	40.94	47.26	53.60	59.94
30°	34.94	41.07	47.38	53.72	60.06
32°	35.06	41.19	47.51	53.85	60.19
34°	35.18	41.32	47.64	53.98	60.32
36°	35.30	41.44	47.76	54.10	60.44
38°	35.42	41.57	47.89	54.23	60.57
40°	35.54	41.69	48.02	54.36	60.70
42°	35.65	41.82	48.14	54.48	60.82
44°	35.77	41.94	48.27	54.61	60.95
46°	35.89	42.07	48.40	54.74	61.08
48°	36.01	42.19	48.52	54.86	61.20
50°	36.13	42.32	48.65	54.99	61.33
52°	36.25	42.45	48.78	55.12	61.46
54°	36.37	42.57	48.90	55.24	61.58
56°	36.50	42.70	49.03	55.37	61.71
58°	36.62	42.83	49.16	55.50	61.84
60°	36.74	42.95	49.28	55.62	61.96
62°	36.86	43.08	49.41	55.75	62.09
64°	36.99	43.20	49.54	55.88	62.22
66°	37.11	43.33	49.66	56.00	62.34
68°	37.23	43.46	49.79	56.13	62.47
70°	37.35	43.58	49.92	56.26	62.60
72°	37.48	43.71	50.04	56.38	62.72
74°	37.60	43.84	50.17	56.51	62.85
76°	37.72	43.96	50.30	56.64	62.98
78°	37.84	44.09	50.43	56.77	63.10
80°	37.97	44.22	50.55	56.89	63.23
82°	38.09	44.34	50.68	57.02	63.36
84°	38.21	44.47	50.80	57.16	63.49
86°	38.33	44.60	50.92	57.27	63.61
88°	38.46	44.72	51.06	57.40	63.74
90°	38.58	44.85	51.19	57.53	63.87
92°	38.70	44.97	51.31	57.65	63.99
94°	38.82	45.10	51.44	57.78	64.12
96°	38.94	45.23	51.57	57.91	64.25
98°	39.07	45.35	51.69	58.03	64.37
100°	39.19	45.48	51.82	58.16	64.50
M. V. per °C.	.0603	.0629	.0634	.0634	.0634

TABLE 4—Copper-Constantan Thermocouples*

E.m.f.s are expressed in microvolts (1 millivolt = 1000 microvolts).
Temperatures are in Deg. Cent.
Cold junction temperature is 0 Deg. Cent.

–Temperatures and Temperature

E μv	−5000	−4000	−3000	−2000	−1000	−0
0	−169.14	−124.46	−87.86	−55.81	−26.82	0
	5.20	*4.01*	*3.42*	*3.05*	*2.79*	*2.60*
100	−174.34	−128.47	−91.28	−58.86	−29.61	−2.60
	5.40	*4.09*	*3.46*	*3.08*	*2.81*	*2.62*
200	−179.74	−132.56	−94.74	−61.94	−32.42	−5.22
	5.64	*4.18*	*3.51*	*3.11*	*2.84*	*2.63*
300	−185.38	−136.74	−98.25	−65.05	−35.26	−7.85
	5.89	*4.28*	*3.57*	*3.15*	*2.86*	*2.65*
400	−191.27	−141.02	−101.82	−68.20	−38.12	−10.50
	6.17	*4.39*	*3.63*	*3.19*	*2.89*	*2.67*
500	−197.44	−145.41	−105.45	−71.39	−41.01	−13.17
	6.51	*4.50*	*3.68*	*3.22*	*2.90*	*2.69*
600	−203.95	−149.91	−109.13	−74.61	−43.91	−15.86
	6.97	*4.61*	*3.74*	*3.26*	*2.93*	*2.71*
700	−210.92	−154.52	−112.87	−77.87	−46.84	−18.57
	7.55	*4.73*	*3.80*	*3.29*	*2.96*	*2.73*
800	−218.47	−159.25	−116.67	−81.16	−49.80	−21.30
		4.87	*3.86*	*3.33*	*2.99*	*2.75*
900		−164.12	−120.53	−84.49	−52.79	−24.05
		5.02	*3.93*	*3.37*	*3.02*	*2.77*
1000		−169.14	−124.46	−87.86	−55.81	−26.82

E μv	7000	8000	9000	10,000	11,000	12,000
0	155.95	175.50	194.62	213.36	231.74	249.82
	1.97	*1.93*	*1.89*	*1.85*	*1.82*	*1.79*
100	157.92	177.43	196.51	215.21	233.56	251.61
	1.97	*1.93*	*1.89*	*1.85*	*1.82*	*1.79*
200	159.89	179.36	198.40	217.06	235.38	253.40
	1.97	*1.92*	*1.88*	*1.85*	*1.82*	*1.78*
300	161.86	181.28	200.28	218.91	237.20	255.18
	1.96	*1.92*	*1.88*	*1.84*	*1.81*	*1.78*
400	163.82	183.20	202.16	220.75	239.01	256.96
	1.96	*1.91*	*1.88*	*1.84*	*1.81*	*1.78*
500	165.78	185.11	204.04	222.59	240.82	258.74
	1.95	*1.91*	*1.87*	*1.84*	*1.81*	*1.78*
600	167.73	187.02	205.91	224.43	242.63	260.52
	1.95	*1.91*	*1.87*	*1.83*	*1.80*	*1.77*
700	169.68	188.93	207.78	226.26	244.43	262.29
	1.94	*1.90*	*1.86*	*1.83*	*1.80*	*1.77*
800	171.62	190.83	209.64	228.09	246.23	264.06
	1.94	*1.90*	*1.86*	*1.83*	*1.80*	*1.77*
900	173.56	192.73	211.50	229.92	248.03	265.83
	1.94	*1.89*	*1.86*	*1.82*	*1.79*	*1.77*
1000	175.50	194.62	213.36	231.74	249.82	267.60

*Standard calibration curve for copper-constantan thermocouple giving the temperature and temperature differences for every 100 microvolts. Fixed junction is at 0°. For use in conjunction with a deviation curve determined by calibration of the particular couple at some of the following fixed points:

	Degrees C.	Micro-volts μv		Degrees C.	Micro-volts μv
Oxygen, boiling point........	−182.98	5,258	Naphthalene, boiling point...	217.95	10,248
Carbon dioxide, boiling point..	−78.5	2,719	Tin, melting point...........	231.9	11,009
Mercury, melting point.......	−38.88	1,426	Benzophenone, boiling point	305.9	13,203
Water, boiling point..........	100.00	4,276	Cadmium, melting point.....	320.9	16,083

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*Temperatures and Temperature Differences for Copper-Constantan Thermocouple.

TABLE 4 (*Continued*)

*Differences for Copper-constantan Thermocouple**

0	1000	2000	3000	4000	5000	6000
0	25.27	49.20	72.08	94.07	115.31	135.91
2.59	*2.45*	*2.33*	*2.23*	*2.16*	*2.09*	*2.03*
2.59	27.72	51.53	74.31	96.23	117.40	137.94
2.57	*2.43*	*2.32*	*2.23*	*2.15*	*2.08*	*2.02*
5.16	30.15	53.85	76.54	98.38	119.48	139.96
2.56	*2.42*	*2.31*	*2.22*	*2.14*	*2.08*	*2.02*
7.72	32.57	56.16	78.76	100.52	121.56	141.98
2.55	*2.41*	*2.30*	*2.21*	*2.14*	*2.07*	*2.01*
10.27	34.98	58.46	80.97	102.66	123.63	143.99
2.53	*2.40*	*2.30*	*2.20*	*2.13*	*2.06*	*2.01*
12.80	37.38	60.76	83.17	104.79	125.69	146.00
2.52	*2.39*	*2.28*	*2.20*	*2.12*	*2.06*	*2.00*
15.32	39.77	63.04	85.37	106.91	127.75	148.00
2.51	*2.38*	*2.27*	*2.19*	*2.11*	*2.05*	*2.00*
17.83	42.15	65.31	87.56	109.02	129.80	150.00
2.49	*2.36*	*2.27*	*2.18*	*2.10*	*2.04*	*1.99*
20.32	44.51	67.58	89.74	111.12	131.84	151.99
2.48	*2.35*	*2.25*	*2.17*	*2.10*	*2.04*	*1.99*
22.80	46.86	69.83	91.91	113.22	133.88	153.97
2.47	*2.34*	*2.25*	*2.16*	*2.09*	*2.03*	*1.98*
25.27	49.20	72.08	94.07	115.31	135.91	155.95

13,000	14,000	15,000	16,000	17,000	18,000	19,000
267.60	285.13	302.42	319.49	336.36	353.08	369.61
1.76	*1.74*	*1.72*	*1.70*	*1.68*	*1.66*	*1.64*
269.36	286.87	304.14	321.19	338.04	354.74	371.25
1.76	*1.74*	*1.71*	*1.69*	*1.68*	*1.66*	*1.64*
271.12	288.61	305.85	322.88	339.72	356.40	372.89
1.76	*1.74*	*1.71*	*1.69*	*1.68*	*1.66*	*1.64*
272.88	290.35	307.56	324.57	341.40	358.06	374.53
1.76	*1.73*	*1.71*	*1.69*	*1.67*	*1.66*	*1.64*
274.64	292.08	309.27	326.26	343.07	359.72	376.17
1.76	*1.73*	*1.71*	*1.69*	*1.67*	*1.65*	*1.63*
276.40	293.81	310.98	327.95	344.74	361.37	377.80
1.75	*1.73*	*1.71*	*1.69*	*1.67*	*1.65*	*1.63*
278.15	295.54	312.69	329.64	346.41	363.02	379.43
1.75	*1.72*	*1.70*	*1.68*	*1.67*	*1.65*	*1.63*
279.90	297.26	314.39	331.32	348.08	364.67	381.06
1.75	*1.72*	*1.70*	*1.68*	*1.67*	*1.65*	*1.63*
281.65	298.98	316.09	333.00	349.75	366.32	382.69
1.74	*1.72*	*1.70*	*1.68*	*1.67*	*1.65*	*1.63*
283.39	300.70	317.79	334.68	351.42	367.97	384.32
1.74	*1.72*	*1.70*	*1.68*	*1.66*	*1.64*	*1.63*
285.13	302.42	319.49	336.36	353.08	369.61	385.95

EXPERIMENT NO. 4

The Calibration of a Resistance Thermometer

REFERENCES

Foote, Fairchild, and Harrison, Pyrometric Practice, National Bureau of Standards Technologic Paper No. 170, p. 127-134, 278-281 (1921).

Burgess, The International Temperature Scale, National Bureau of Standards, Research Paper No. 22 (1928).

The resistance thermometer consists essentially of a coil of pure platinum wire, the resistance of which may be accurately measured by a suitable Wheatstone bridge arrangement.

For temperature measurements within its range, the platinum resistance thermometer may be made a most precise and dependable instrument. In fact, the extreme precision that it is possible to attain with this instrument has led to its adoption as a working standard for defining the International Temperature Scale from −190° C. to 660° C.

The importance of the resistance thermometer in industrial work is declining due to the many advantages possessed by the thermoelectric type of pyrometer, but the resistance thermometer still easily maintains its preeminent position as a precise means of determining temperatures in laboratory investigations.

Object. To calibrate a platinum resistance thermometer by determining its resistance at the melting point of ice, the boiling point of water, and the boiling point of sulfur.

THEORY

Between −190° C. and 0° C., the International Temperature Scale is defined by the platinum resistance thermometer calibrated at the boiling point of oxygen, the melting point of ice, the boiling point of water, and the boiling point of sulfur. Interpolation between −190° C. and 0° C. is accomplished by the following equation

$$R_t = R_0[1 + At + Bt^2 + C(t - 100)t^3] \tag{1}$$

R_t represents the resistance measured, R_0 the resistance at 0° C., t the temperature in degrees Centigrade, while A, B, and C are constants which are evaluated from the calibration data.

Between 0° C. and 660° C., the International Temperature Scale is defined by the platinum resistance thermometer calibrated at the melting point of ice, the boiling point of water, and the boiling point of sulfur.

Interpolation between these fixed points is accomplished by the following equation.

$$R_t = R_0[1 + At + Bt^2] \tag{2}$$

The direct utilization of this equation presents the disadvantage of requiring tedious calculations for the evaluation of the constants A and B. Furthermore, once the numerical values of the constants A and B have been determined, the equation is not in very usable form inasmuch as we have a quadratic in t. If a resistance is measured and the corresponding temperature is to be determined, a difficult solution of the quadratic is required. One alternative is to assume a series of values of t, and after solving the equation for values of R_t, to plot a calibration curve, R_t versus t. This procedure is open to the objection of demanding an extremely long curve in order that temperatures may be evaluated to the degree of precision warranted by the precision of the resistance measurements.

The difficulties pointed out in the preceding paragraph may be avoided by utilizing the method of calculation first advocated by H. L. Callendar, which is along the following general lines: If the temperature-resistance relation were exactly linear, temperatures could be evaluated from resistance readings by the following equation:

$$\text{Temperature} = \frac{R_t - R_0}{R_{100} - R_0} \times 100 \tag{3}$$

where R_t is the measured resistance, and R_0 and R_{100} are the respective resistances at 0° and 100° C. The resistances at 0° C. and 100° C. are determined in calibrating the thermometer. Actually, of course, the resistance-temperature relationship is not linear, and consequently the temperatures thus computed are fictitious rather than true temperatures. The fictitious temperatures defined by the above equation are commonly referred to as "platinum temperatures" and are designated by the symbol "*pt*."

$$pt = \frac{R_t - R_0}{R_{100} - R_0} \times 100 \tag{4}$$

After the numerical value of the fictitious temperature "*pt*" is evaluated for a particular resistance, it becomes necessary to apply a correction in order to convert the fictitious temperature to the true temperature. This is accomplished by the use of the following equation, where t represents true temperature, "*pt*" the fictitious or "platinum temperature," and "*d*" a constant determined from the calibration data for the thermometer.

$$(t - pt) = d\left[\frac{t}{100} - 1\right]\frac{t}{100} \tag{5}$$

By utilizing equation (5) a plot of $(t - pt)$ versus "pt" is prepared, which enables one to apply rapidly the correction needed to convert the fictitious temperature into true temperature.

From the foregoing, it should be apparent that the determination of the temperature corresponding to a given resistance reading of a platinum resistance thermometer resolves itself into three steps: (1) Determine the value of "pt" from equation No. 4. This can be done rapidly and accurately with a table of logarithms. (2) Consult the correction curve based on Equation No. 5, in which $(t - pt)$ is plotted versus "pt." (3) Evaluate the true temperature from the value of "pt" and the value of $(t - pt)$.

It is obvious that before the above procedure can be followed, it is necessary to determine R_0, R_{100}, and the numerical value of "d" in Equation No. 5 for the particular thermometer under consideration. In this experiment, R_0 and R_{100} are determined, as well as the resistance at the boiling point of sulfur. As will be explained more fully later, the latter value is utilized for evaluating the constant "d." Once the numerical value of "d" has been obtained, it will be possible to plot the $(t - pt)$ versus "pt" curve, and then it will be possible to evaluate rapidly the temperature corresponding to any particular resistance.

If equations 4 and 5 are combined, an equation having the form of No. 2 will be obtained. Hence, the use of equations 4 and 5 in computational work is sound in principle and does not violate the specification that the temperature scale shall be defined by an equation having the form of No. 2.

Lead Wire Compensation. The resistance thermometer ordinarily consists of a small coil of platinum wire wound on thin mica supports. The coil is mounted in the closed end of a long protection tube of glass, quartz, or porcelain. Leads from the coil extend to binding posts at the opposite end of the tube. In order that the readings of the instrument may be independent of the depth of immersion of the tube, variations in the resistance of these leads must be either minimized or compensated for.

In the thermometers used in this experiment, the Callendar, or "dummy lead" method of compensation is used. Mounted beside the leads connected to the coil are two "dummy" leads, identical with the first in every respect. The ends of these leads next to the coil are connected together, forming a closed circuit of the two. This loop is then connected into the variable arm of the Wheatstone bridge adjacent to that containing the coil and its pair of leads. It is apparent that a change in temperature of one pair of leads will be accompanied by a similar change of both temperature and resistance of the other. If the two ratio arms of the bridge are equal these two effects will balance each other and the setting of the variable arm will remain independent of such changes. The circuit employed in such compensation is shown on the accompanying wiring diagrams.

The Calibration Points

The Ice Point: The melting point of finely divided pure ice at atmospheric pressure is taken as exactly zero degrees Centigrade. The changes in the melting point which are produced by atmospheric pressure variations are ordinarily negligible. Care must be taken that the ice is pure and that the thermometer is immersed in it to a depth sufficient to render negligible the conduction of heat along the tube to the coil.

The Boiling Point of Water: The boiling point of pure water is represented by the following expression, where "p" is the prevailing atmospheric pressure in mm. of mercury at 0° C. and at standard gravity.

$$t^\circ \text{ C.} = 100.00 + 0.0367(p - 760) - 0.000023(p - 760)^2 \qquad (6)$$

In determining the true atmospheric pressure the reading of the ordinary brass-scale barometer must be corrected to 0° C. and standard gravity. The column height is corrected to 0° C. by the following equation.

$$H_0 = R - \left[\frac{0.0001634t}{1 + 0.0001818t}\right] R \qquad (7)$$

H_0 represents the column height at 0° C., t the temperature of the barometer in degrees Centigrade, and R the barometer reading in millimeters of mercury. The reduction to standard gravity (980.665 cm./sec.^{2}) is accomplished by the following equation:

$$p = H_0 - H_0 \left[\frac{980.665 - g_l}{980.665}\right] \qquad (8)$$

H_0 represents the column height in mm. of mercury, corrected for temperature, as evaluated by Equation (7).

g_l = local value for acceleration of gravity, expressed as cm./sec.^{2}

p = column height at 0° C. and standard gravity, in millimeters of mercury.

In case the local value for g is unknown, the gravity correction may be estimated from tables wherein the gravity correction is given as a function of latitude and altitude. Such tables are printed in many handbooks. (For example, in the Handbook of Chemistry and Physics, published by the Chemical Rubber Co.)

Thus the true temperature corresponding to the boiling point of water under any pressure conditions may be calculated. In using the steam point for calibrations care must be taken that the water is pure and that the pressure at its surface is not appreciably above atmospheric. The surfaces surrounding the thermometer must also be at the temperature of the steam to prevent radiation interchange between them and the thermometer. Similarly the depth of immersion must be sufficient to prevent conduction of heat along the tube from introducing an error. The Regnault hypsometer is designed to fulfill these requirements and is used in the calibration.

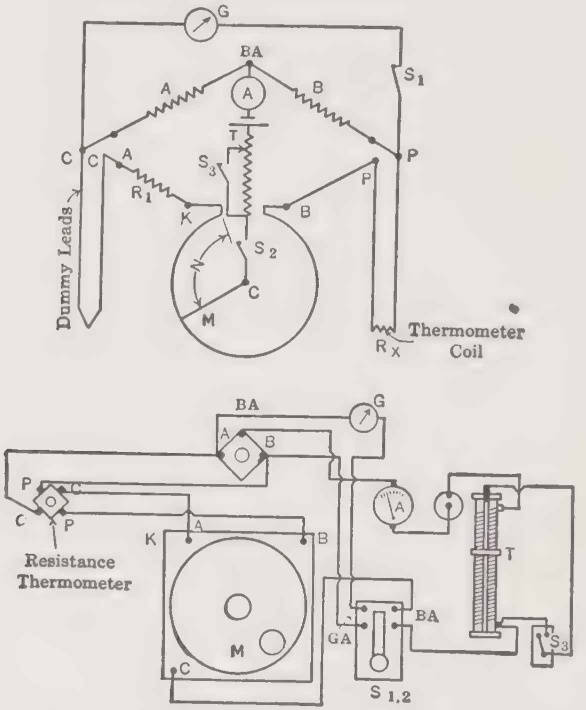

FIG. 13.—Thermometric resistance bridge.

The Sulfur Boiling Point: The boiling point of sulfur is represented by the following expression:

$$t^\circ \text{ C.} = 444.60 + 0.0909(p - 760) - 0.000048(p - 760)^2 \qquad (9)$$

In using the boiling point of sulfur as a calibration point, the same precautions must be observed as in the use of the steam point. In addition, great care must be exercised to prevent the superheating of the sulfur vapor. The Bureau of Standards has devised an apparatus for the accurate determination of this point. (Reference No. 1, pp. 278-282.)

Procedure. In handling the resistance thermometers care must be exercised, since they are very fragile and expensive.

The resistance of the thermometer is to be measured by either of the two Wheatstone bridge arrangements shown in Figs. 13 and 14.

Operating Directions for Fig. 13.

With the galvanometer disconnected, close switch S_3, depress key $S_{1,\,2}$, and adjust contact T until the milliammeter A shows a reading of 10.

With the galvanometer connected in the circuit and *switch S_3 open,* adjust contact M until the galvanometer G shows no deflection on momentarily depressing key $S_{1,\,2}$. Do not hold the key closed longer than necessary. A final adjustment may then be made by closing switch S_3 to increase the response of the galvanometer.

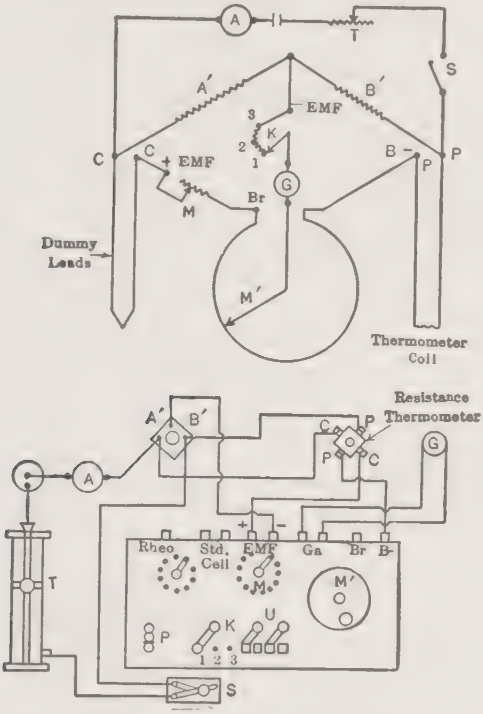

Fig. 14.—Thermometric resistance bridge employing the Leeds and Northrup Type K Potentiometer.

The bridge reading N may now be directly obtained. The resistance of x, in ohms, is then:

$$R_x = 2 \times N \times 0.007082 \text{ or } 0.014164\,N;$$

where N is the bridge reading in small divisions.

Operating Directions for Fig. 14.

Set contact M on point "1"; plug P in position "1.0"; and switch U in the "E.M.F." position.

Depress key S and adjust rheostat T until ammeter A shows a reading of 10 milliamperes.

Set key K above position "1."

Momentarily close keys S and K and adjust contact M' until galvanometer G shows no deflection. Key S should always be closed an instant before K.

For closer adjustment of M', key K may be moved to position "2" and then to position "3" which produces maximum galvanometer response.

The graduations on contact M' represent the resistance of the thermometer coil in ohms, one revolution of the contact being equivalent to one ohm.

The Ice Point: The thermometer is mounted in a large thermally insulated glass cylinder. The end of the thermometer protection tube should be about 2 inches above the bottom of the cylinder. The cylinder is filled to the top with finely chipped ice. After a few minutes the reading of the bridge is recorded at one minute intervals until five successive readings are identical.

The Steam Point: For the determination of the steam point the lower compartment of the hypsometer is filled about two thirds full of distilled water and supported on a ring stand. The resistance thermometer is inserted through the opening in the top and supported so that the tip of the tube is just at the top of the lower compartment. A cloth should be packed around the thermometer where it enters the hypsometer to prevent steam from rising along the tube. A beaker is placed under the steam outlet. The water is then brought to a vigorous boil so that steam is continuously emerging from the outlet. However, heating should not be stronger than necessary to just maintain such a flow. Readings of the bridge are then recorded at one minute intervals until five successive readings are identical. The height and temperature of the barometer are recorded during this period.

The Sulfur Point: An electrically heated sulfur boiling apparatus is available. In operating this piece of equipment, the current recommended by the instructor should not be exceeded. The Pyrex glass tube containing the sulfur should be filled to a depth of not less than 4 inches. The thermometer is equipped with a metal radiation shield and should be supported in the apparatus so that it is centered and the tip of its bulb is about 2 inches above the level of the molten sulfur. The top of the boiling tube should be covered with a loosely fitting sheet of asbestos to reduce oxidation of the sulfur vapors. After the sulfur has come to a boil, sufficient current should be passed through the heating coil to keep the line of condensation in sight somewhat above the top of the insulating jacket surrounding the glass tube. Readings of the bridge are then recorded at one minute intervals until five successive readings are alike. The barometer is again read, and the temperature at this barometer reading is noted.

After a satisfactory set of data has been obtained, the heating current is shut off and the apparatus is allowed to stand five or ten minutes with the purpose of permitting the boiling to subside. The thermometer is then removed and rapidly transferred to an empty bottle, in which the sulfur that inevitably ignites on removal from the boiling tube will soon be extinguished. Care should be taken not to break the protection tube in making this transfer.

When the boiling apparatus is no longer to be used the top of the tube should be covered with a sheet of asbestos and the whole apparatus inclined at an angle of about 30° with the horizontal. This is necessary in order that the sulfur may solidify along the sides of the tube and not break it on subsequent heating.

REPORT

Before beginning the calculations, the student should estimate the precision of temperature measurements made with the equipment employed, assuming that bridge readings can be duplicated under steady conditions within $\pm$ 0.1 scale division. All of the calculations must be carried out so that the full accuracy of the data is realized. In general, an analysis of the situation will show that the slide rule is unsuited for carrying out the major part of the work, though it may of course be used to evaluate terms of relatively small magnitude. This calculation of the precision of temperature measurements should be the first item on the calculation sheet.

By assuming that the value of "d" in equation No. 5 is equal to 1.5 the "platinum temperature" at the steam point may be calculated. The error introduced by this assumption may ordinarily be neglected because of the very small difference between "t" and "pt" at the steam point. In addition, the value of "d" for pure platinum is generally between 1.48 and 1.52. If the final value of "d" as found from the sulfur point is widely different from 1.5 a second approximation should be made, substituting the computed value for "d" in the calculation of "pt" at the steam point.

The "platinum temperature" at the sulfur point may now be calculated from equation No. 4. The true value of "d" is then determined by equation No. 5, using the "t" and "pt" for the sulfur point.

Before proceeding further, the accuracy of the calculations should be checked, which may be done as follows. From the calculated value of "*d*," and the known temperature at the sulfur boiling point, "*pt*" at the sulfur point is calculated by means of Equation No. 5. This value of "*pt*" is then substituted in Equation No. 4, and the equation is solved for R_t at the sulfur point. If this calculated value of R_t checks the experimentally determined value exactly, the calculations have been accurately made. Failure to check does not indicate experimental error; it shows poor arithmetic. If an exact check is not obtained the calculations must be corrected. The check on the accuracy of the value of "*d*" should be clearly shown on the computation sheet.

The next step is the construction of the so called "difference curve," which is a plot of $(t - pt)$ versus "*pt*." The numerical value of "*d*" which was just calculated is inserted into Equation No. 5. A series of values of "*t*" are assumed, and the corresponding values of "*pt*" calculated. A table is prepared, in which values of $(t - pt)$ are tabulated against values of "*pt*." These figures are used to plot the "difference curve," $(t - pt)$ versus "*pt*."

The value of $(t - pt)$ is zero at both 0° C. and 100° C., and exhibits a minimum between these two temperatures. That this minimum comes at exactly 50° C. (true temperature) may be shown by differentiating equation No. 5, setting the slope equal to zero, and solving for *t*.

In plotting the $(t - pt)$ versus "*pt*" curve, the scale should be great enough so that the full accuracy of the experimental data is realized.

SAMPLE DATA AND CALCULATIONS

Thermometer No. 243

Wiring as shown in Fig. 13.

	Bridge Reading
Ice point	185.5
Steam point	255.8
Sulfur point	483.0
Barometer reading	736.1 mm. Hg.
Barometer temperature	21° C.

Since the bridge readings are directly proportional to resistance it is permissible in all of the following calculations to employ bridge readings directly, without converting into ohms.

Estimate of Precision of Temperature Measurements:

Between the steam and ice points, 70.3 scale divisions are covered. The temperature difference is approximately 100 deg. C. If the bridge reading is in error ± 0.1 scale division, the error in temperature measurement is

$$\frac{0.1}{70.3} \times 100 = 0.14 \text{ deg. C.}$$

Accordingly, temperatures should not be expressed beyond the first decimal place.

Corrected Barometric Pressure:

H_0 by Equation (7) = 733.6 mm. Hg.
p by Equation (8) = 733.4 mm. Hg.
(g_t = 980.365 cm./sec.^{2})

Temperature of Steam Point:
By equation (6), t_{st} = 99.01 deg. C.

Temperature of Sulfur Point:
By equation (9), t_s = 442.15 deg. C.

Evaluation of Bridge Reading at Exactly 100° C.:
Assume $d = 1.5$ and substitute in equation (5)

$$99.01 - pt = 1.5\ [.9901-1]\ .9901$$

pt at steam point = 99.02
Next substitute in equation (4)

$$99.02 = \frac{255.8 - 185.5}{R_{100} - 185.5} \times 100$$

$R_{100} = 256.5$

Evaluation of "d" from Data at Sulfur Point:
Using equation (4)

$$pt = \frac{483.0 - 185.5}{256.5 - 185.5} \times 100 = 419.0$$

Using equation (5)

$$442.2 - 419.0) = d\ (4.422 - 1)\ 4.422$$

$d = 1.532$

This value of "d" is near enough to the previously assumed value of 1.50 for d so that a second approximation is not needed.

Data for Plotting Difference Curve:

t	$t/100$	$\frac{t}{100} - 1$	$\left[\frac{t}{100} - 1\right] \frac{t}{100}$	$(t - pt)$	pt
0° C.	0.00	−1.00	0.00	0.00	0.0° C.
20	0.20	−0.80	−0.16	−0.2	20.2
40	0.40	−0.60	−0.24	−0.4	40.4
50	0.50	−0.50	−0.25	−0.4	50.4
60	0.60	−0.40	−0.24	−0.4	60.4
80	0.80	−0.20	−0.16	−0.2	80.2
100	1.00	−0.00	0.00	0.00	100.0
150	1.50	0.50	0.75	+1.1	148.9
200	2.00	1.00	2.00	3.1	196.1
250	2.50	1.50	3.75	5.7	244.3
300	3.00	2.00	6.00	9.2	291.8
350	3.50	2.50	8.75	13.4	336.6
400	4.00	3.00	12.00	18.4	381.6
442.2	4.422	3.422	15.13	23.2	419.0
450	4.50	3.50	15.75	24.1	425.9
500	5.00	4.00	20.00	30.6	469.4
550	5.50	4.50	24.75	37.9	512.1
600	6.00	5.00	30.00	46.0	554.0
650	6.50	5.50	35.75	54.7	595.3

Values of "*pt*" and $(t - pt)$ from the above table are used to plot the difference curve.

Method of Converting Resistance Readings into Temperature:

If the bridge reading is 375.6 scale divisions, what is the temperature?

$$pt = \frac{375.6 - 185.5}{256.5 - 185.5} \times 100 = 267.7 \text{ (Evaluated with 4 place logarithms)}$$

Referring to the difference curve (Fig. 15), we see that for a "*pt*" value of 267.7, $(t - pt)$ equals 7.3 deg. $t = 267.7 + 7.3 = 275.0$ deg. C.

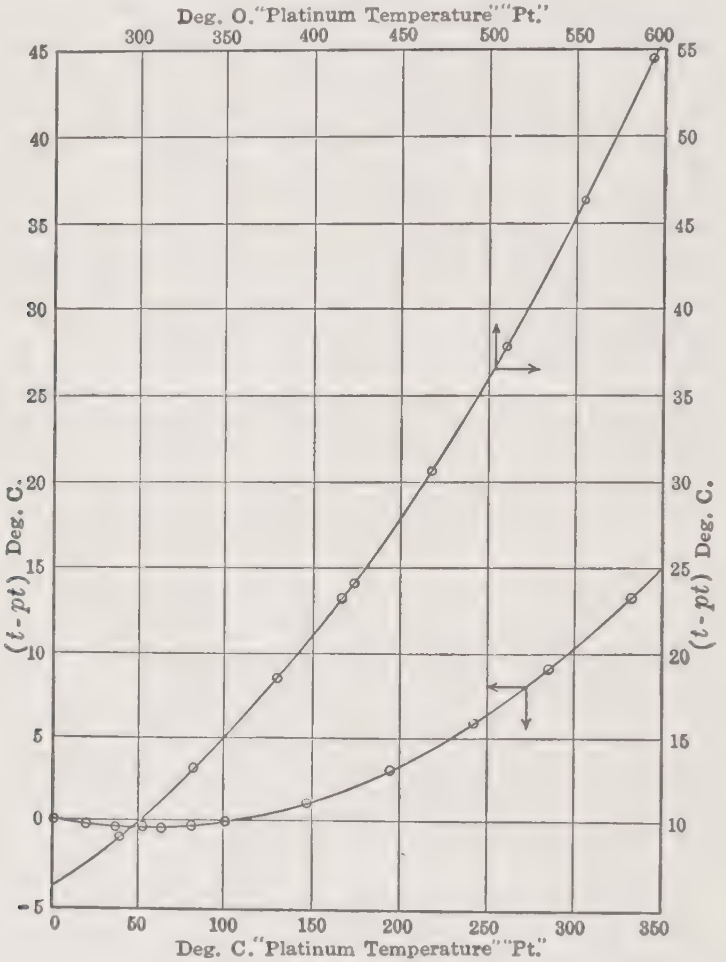

FIG. 15.—Difference curve.

EXPERIMENT NO. 5

The Secondary Standardization of a Disappearing Filament Optical Pyrometer

REFERENCE

Foote, Fairchild, and Harrison, *Pyrometric Practice*, National Bureau of Standards Technologic Paper No. 170, pp. 258-273.

To eliminate the necessity of performing a tedious and difficult primary calibration, methods have been developed whereby an optical pyrometer may be quickly standardized by comparison with another instrument of known calibration. The most rapid and satisfactory of these methods is that employing a broad filament tungsten lamp as a source of radiation.

Object. To calibrate an optical pyrometer against a standard by means of a broad filament lamp.

THEORY

Secondary Standardization. An optical pyrometer may be subjected to secondary calibration by sighting it on a source, the temperature of which is measured by another standard instrument of the same type. If both instruments utilize the same wave length of light for comparison, it is not necessary that the temperature source be a black-body. If the emissivity of the source is less than 1.0, the standard instrument, which is calibrated to indicate black-body temperatures, will indicate the apparent temperature of the source, or the temperature to which a black body would have to be heated to emit radiations of the same intensity as the source. The instrument being calibrated will be sighted on the same source and the effect will be the same as if the two instruments were sighted on a black body of somewhat lower temperature.

A very satisfactory source for the comparison of optical pyrometers is the broad filament tungsten lamp. The most accurate method for using such a lamp is to sight the two pyrometers to be compared on the same spot on the filament, from the same angle. Readings of both are then recorded at various lamp currents. Great care must be exercised to sight both of the pyrometers on the same part of the filament.

Where a large number of secondary calibrations is to be made, a more rapid method, of generally satisfactory accuracy, is provided by determining the current-black body temperature calibration of the broad-filament lamp. The standard optical pyrometer is used for this purpose, determining the apparent or black body temperatures of some designated part of the filament. An accurate ammeter is used to measure the current in the large lamp. A curve may then be plotted relating the black body temperature of the lamp to the current through it. The pyrometer to be calibrated is sighted on the designated spot of the broad filament and its readings corresponding to various currents in the large lamp are determined and recorded. Such a method of calibration introduces the uncertainty of the calibration of the broad filament lamp, but if this is checked at reasonable intervals the degree of accuracy obtained is within that ordinarily required.

Extension of Range of Optical Pyrometers. The temperature at which the filament of the optical pyrometer lamp is operated should not exceed about 1400° C., otherwise the filament will deteriorate, and the calibration of the lamp will be altered. In using such an instrument for the measurement of high temperatures, it is necessary to reduce the intensity from the source in order that the filament may be adjusted to match it. This reduction may be accomplished by interposing an absorbing medium between the source and the pyrometer lamp. Energy incident upon such a medium will be transmitted in part and absorbed in part. The proportion of the incident energy which is transmitted is known as the "transmission coefficient" of the absorption medium. When such an energy absorbing screen is used before an optical pyrometer, the instrument will indicate some apparent black body temperature which is lower than the true black body temperature. The relation between apparent and true black body temperatures may be deduced by applying Wien's Law.

$$J_\lambda = c_1\lambda^{-5}[e]^{-\frac{C_2}{\lambda S_a}} = Rc_1\lambda^{-5}[e]^{-\frac{C_2}{\lambda S_t}}$$

λ = effective wave length of light transmitted by the colored glass in the ocular
c_1 = a constant
c_2 = 14,320 micron deg. K.
e = base of Naperian logarithms
J_λ = intensity of radiation of wave length λ
R = transmission coefficient of absorption device
S_a = apparent black body temperature of source in absolute degrees
S_t = true black body temperature of source in absolute degrees.

By combining various terms in the above equation, the following working equation may be deduced:

$$\frac{1}{S_t} - \frac{1}{S_a} = \frac{\lambda \log R}{c_2 \log e}$$

The right-hand side of the equation is, for all practical purposes, a constant for a given pyrometer, hence the equation may be written

$$\frac{1}{S_t} - \frac{1}{S_a} = A$$

By means of this expression, it is possible to calculate the complete calibration of an optical pyrometer when used with an absorption screen if the value of the constant "*A*" and the original calibration of the instrument without the absorption screen are known.

Glass Absorbing Screen. The most simple form of absorption screen consists of a piece of colored glass mounted before the pyrometer lamp. When using such an absorption device, the value of the constant "*A*" is determined by sighting the pyrometer on a source of constant temperature and making observations with and without the screen in position before the pyrometer lamp. From the two current values determined under these conditions, the true and apparent black body temperatures of the source are read from the calibration curve, and then the numerical value of the constant "*A*" may be computed. It is desirable to make such observations at two or three different source temperatures, and to average the values of "*A*" thus determined. From the mean value of "*A*" the complete calibration of the pyrometer with the screen in place may be calculated from the original calibration. The assumption that "*A*" is constant is slightly in error because of the fact that the pyrometer does not use perfectly monochromatic light. For ordinary practical work corrections for such errors may be neglected.

Rotating Sectored Disc. For laboratory work a very satisfactory absorption screen is obtained by interposing between the pyrometer lamp and the source a rotating opaque disc from which a definite sector has been removed. The disc must be rotated at a speed above that which produces flickering visible to the eye. The transmission coefficient of a screen so produced is equal to the proportion of the total area of the disc which is represented by the removed sector. Thus a pyrometer may be provided with a set of these discs, the transmission coefficients of which are known from their construction. If the effective wave length of light transmitted by the ocular is known, as well as the transmission coefficient of the sectored disc (from the proportion of the total area removed) it is possible to calculate "*A*" for the assembly. However, if either or both of the required items of data are lacking, it becomes necessary to evaluate "*A*" by experiment, just as when using the glass absorption screen before the pyrometer lamp. Once "*A*" has been determined, the calibration curve for the pyrometer with the disc in use may be calculated from the original calibration.

In general, the determination of the constant "A" for an absorption device (colored glass or sectored disc) requires careful work. The calibration curve of the pyrometer without the absorption device must be accurate, and in making the given pair of observations required for the evaluation of "A" it is necessary that the source temperature be maintained absolutely constant. Any inaccuracy in the numerical value of "A" will introduce errors into the computed high range calibration of the optical pyrometer. In general, if the calibration data available for the standard pyrometer permit, it is desirable to calibrate the unknown instrument both with and without its absorption device, by direct comparison with the standard. This procedure is less likely to introduce error than if the low range alone is determined by direct comparison with the standard, and the high range calibration then calculated by means of an experimentally determined value of "A" for the absorption device on the unknown.

Procedure. The standard optical pyrometer is of the disappearing filament type, and is equipped with a lamp calibrated by the National Bureau of Standards. A glass absorption screen, which can be swung into position between the pyrometer lamp and the hot source, is provided in order to make it possible to measure temperatures in excess of 1400° C. Two curves are supplied for the standard pyrometer, one to be used when the absorption screen is not used, and the other to be used when the absorption screen is in position before the pyrometer lamp.

The optical pyrometer to be calibrated is also of the disappearing filament type, but instead of having a glass absorption screen for extending the range, a set of sectored discs is provided. Any one of these discs may be selected and mounted on the shaft which rotates the disc before the pyrometer objective lens. In this experiment, a disc having one-tenth of its area removed is to be used. The disc is rotated by means of a small electric motor connected to a 6-volt storage battery. When in use, the disc should be rotated at a speed sufficient to eliminate visible flickering. Whenever a disc is not in actual use, it should be removed from the pyrometer. Failure to remove the disc may result in partial obstruction of the objective lens, which would introduce error into the results.

The broad filament tungsten lamp that is to serve as the hot source is connected to the 110-volt power supply through a suitable current controlling device, and in series with an ammeter.

Before starting the experimental work, care should be taken to see that the glass of the broad filament lamp is clean, and also that the objective and ocular lenses of both pyrometers are clean. Care should also be taken to see that the red glass is in position in the ocular otherwise monochromatic light will not be used. The ocular is to be brought to such a position that the pyrometer lamp filament is in sharp focus.

Both pyrometers are now directed at the tungsten broad filament, taking care to sight both instruments on exactly the same reference spot of the filament. The two pyrometers should be so placed that the two telescope tubes make the same angle with a perpendicular from the tungsten filament. In order to keep this angle small, the two pyrometers are placed as close together as possible. Each telescope must be sharply focused on the broad filament. When all adjustments have been made, an observer should see a sharp image of the pyrometer lamp filament superimposed on a sharp image of the tungsten broad filament. As indicated before, both optical pyrometer lamp filaments must fall on exactly the same spot of the image of the tungsten broad filament.

The general procedure is to make observations with the standard pyrometer and with the pyrometer being calibrated, at broad filament lamp currents ranging from 7.5 to 14 amperes in steps of ½ ampere. In working with both pyrometers, observations are to be made without the absorption device up to and including a black body temperature of approximately 1400° C. At that temperature and beyond, it is necessary to use the absorption device on each pyrometer in order that the pyrometer lamp filaments will not be damaged.

In making observations, the current through the pyrometer lamp filament is adjusted until, to the eye, the filament tip has the same brilliance as the hot source. When this adjustment is achieved, the filament tip is indistinguishable from the image of the hot source. At least three readings should be taken and averaged. Duplicate observations should check within 3 milliamperes.

REPORT

1. Plot the calibration curve of the pyrometer, both with and without the absorption device.
2. Plot the calibration curve of the broad filament tungsten lamp.
3. The correct value of "*A*" for the glass absorption screen on the standard pyrometer is -0.000116. Compute the value of "*A*" for the standard on the basis of the observations made at a given temperature, both with and without the absorption screen.
4. Compute the value of "*A*" for the sectored disc used with the optical pyrometer that was calibrated. Values are to be calculated at pyrometer lamp currents near the high end, near the low end, and near the middle of the range covered in the high temperature calibration of the instrument.

SAMPLE DATA AND CALCULATIONS

Standard Pyrometer:

Telescope No. 76468
Lamp No. N.B.S. 708
Absorption Screen No. 613

Pyrometer Being Calibrated:
Telescope No. U.W. 101-A49
Lamp No. 106
Sectored Disc Used: 1/10 area removed.

Table of Data

Broad Filament Lamp Current, Amperes	Standard Optical Pyrometer		Black Body Temperature of Broad Filament	Optical Pyrometer Being Calibrated	
	Position of Glass Absorption Screen	Pyrometer Lamp Current, Milliamperes		Use of Sectored Disc	Pyrometer Lamp Current, Milliamperes
7.5	Out	359	941° C.	Not used	339
8.0	Out	382	1010	Not used	369
8.5	Out	412	1092	Not used	402
9.0	Out	447	1175	Not used	437
9.0	In	366	1170		
9.5	Out	473	1235	Not used	470
9.5	In	381	1233		
10.0	Out	504	1299	Not used	507
10.0	In	397	1296		
10.5	Out	530	1347	Not used	534
10.5	In	409	1342		
11.0	Out	555	1391	Not used	565
11.0	In	422	1388	Used	445
11.5	In	436	1436	Used	465
12.0	In	449	1480	Used	483
12.5	In	460	1513	Used	499
13.0	In	473	1555	Used	517
13.5	In	484	1590	Used	534
14.0	In	497	1627	Used	554

The numerical value of "*A*" for the glass screen may be computed from observations made at broad filament lamp currents of 9, 9.5, 10, 10.5, and 11 amperes.

Broad Filament Lamp Current	True Black Body Temperature	Apparent Black Body Temperature	"*A*"
9.0 amps.	1175° C.	961° C.	−0.000119
9.5	1235	1007	−0.000118
10.0	1299	1054	−0.000118
10.5	1347	1086	−0.000119
11.0	1391	1118	−0.000118

Ave. −0.000118

The numerical value of "*A*" for the sectored disc with the optical pyrometer that was calibrated may be computed at pyrometer lamp currents of 450, 500, and 550 milliamperes.

Pyrometer Lamp Current, Milliamperes	True Black Body Temperature	Apparent Black Body Temperature	"*A*"
450	1400° C.	1195° C.	−0.000084
500	1517	1286	−0.000083
550	1619	1367	−0.000082
			Ave. −0.000083

INSTALLATION OF THERMOCOUPLES

The installation of thermocouples for temperature measurements in chemical engineering operations is thought to be worthy of comment, as faulty installation of the couples means incorrect information as to the temperature at the location where the temperature is desired.

An example selected to illustrate this point is a thermocouple installed in a condenser tube where a study is to be made of the film coefficients of the condensing vapor and of the cooling liquid.

Manifestly, the temperatures desired are the surface temperatures of the exterior and of the interior tube wall. Some years past the opinion was held that these surface temperatures could be determined with accuracy by placing a very thin couple on the wall surface, preferably by plating the couple on, and over a considerable area. These methods were found to be unsatisfactory and were soon abandoned.

The method recommended at present, and followed by the outstanding experimenters in this field is to locate the thermocouple junction at some predetermined point in the tube wall, preferably at the midpoint from interior to exterior, then calculate the surface temperatures, using the formula: $Q = kA\Delta t$. From the midpoint *out* a certain area exists through which heat is flowing and from the midpoint *in* another definite area exists through which the same quantity of heat is flowing.

The tube wall, at the point of location of the thermocouple and where the temperature is to be taken, should be disturbed as little as possible during the installation, as any disturbances produce variations in temperature at this point as compared with the temperatures at other undisturbed points.

The installation is illustrated (Fig. 16). Two small holes (a) are drilled in the tube wall, meeting equidistant from exterior and interior wall, the hole (b) is then drilled through the wall to the point where the two branches of the thermocouple meet.

The thermocouple wires, in size just small enough so that they may be inserted in the drill hole, are thrust in, one from each side, and when in place are soldered, brazed, or welded there, the method of attaching depending on the metal of the tube and the kind of couples used; chromel-alumel couples in a Monel metal tube, for example, can be attached using ordinary solder as the ordinary solder has practically the same conductivity as the Monel metal. As little solder is used as is required to unite the couples and fill the hole (b). The tube is then warmed at this point and the space around the couple wires in the drill holes, (a), is filled in with Vinalite or Bakelite insulating cement. The thermocouple leads are also insulated with the same or a similar insulating material.

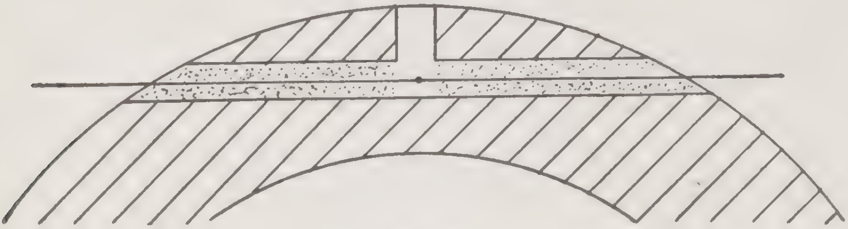

Fig. 16

Some installations are necessary where the temperatures are too high for the cements mentioned. It may be necessary to weld the junction through the hole (b) and use a high temperature cement as the insulating material in the space around the wires in (a).

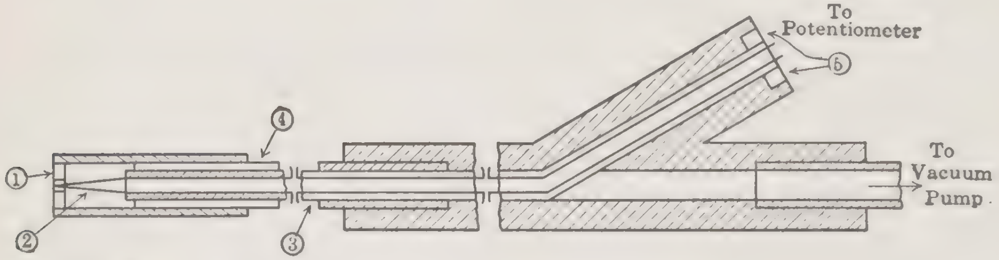

Fig. 17.—High velocity thermocouple.

Various types of thermocouple installations are shown in Experiments 16 to 22. It should never be taken for granted that a thermocouple is correctly installed, and is reading correctly, until it has been calibrated in place. This calibration must be repeated from time to time.

The measurement of gas temperatures in a zone heated by radiation requires a particular type of thermocouple mounting shown in the sketch (Fig. 17).

The principle of operation is: if the gas, the temperature of which it is desired to measure, is drawn past the thermocouple at a sufficiently high rate, the effect of radiant heat will be nil and only the temperature of the gas in the convection current will be measured. This point can be told when further increase in gas velocity induces no temperature change.

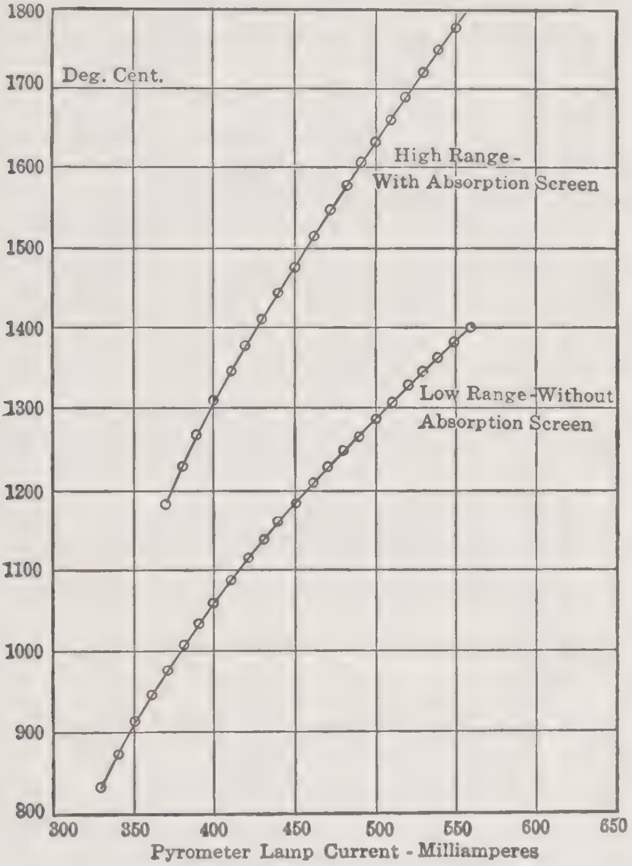

Fig. 18.—Calibration curves for standard optical pyrometer.

The particular features of construction of the thermocouple illustrated are directed towards securing this temperature equilibrium in the minimum time and with the minimum volume of gas passing the thermocouple.

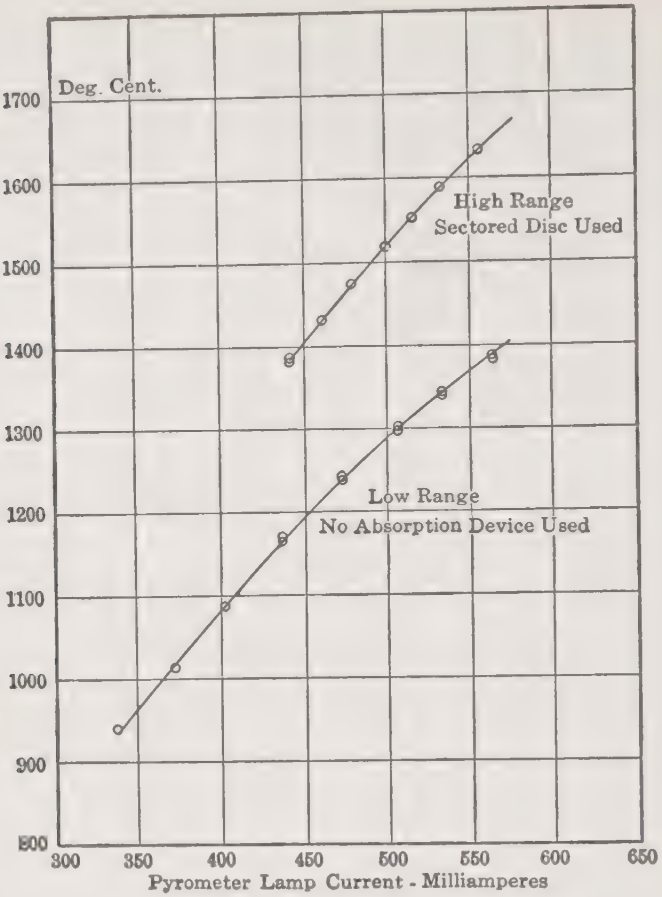

FIG. 19.—Calibration curve of optical pyrometer lamp No. 106.

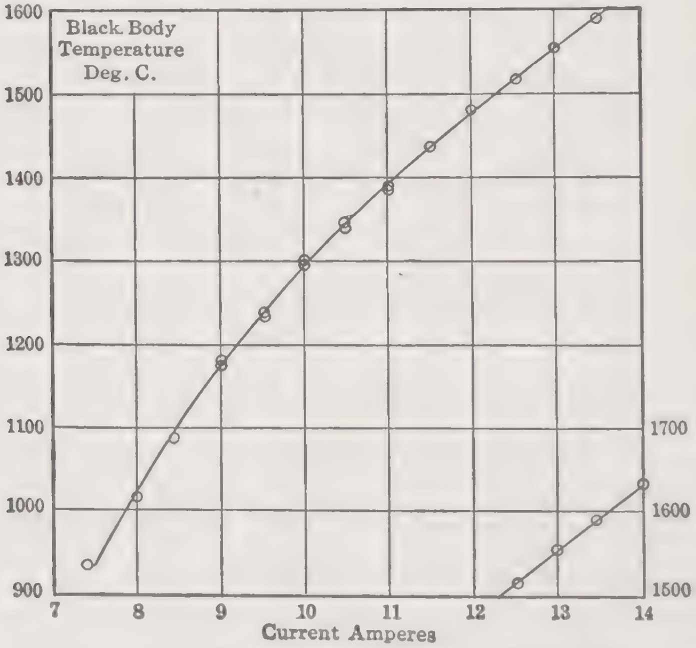

FIG. 20.—Calibration curve of broad filament lamp.

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CHAPTER II

FLOW OF FLUIDS

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HYDROSTATICS AND HYDRODYNAMICS

Hydrostatics and hydrodynamics are the terms given to the consideration of the characteristics of fluids at rest and fluids in motion, respectively. Hydraulics refers generally to the flow and handling of liquids. Aerodynamics refers generally to the flow of gases. The characteristics of liquids and gases are similar in many respects as regards their flow and behavior in engineering equipment, and they will be considered together, except for certain differences which will be pointed out.

The principal interest of the chemical engineer in hydrostatics is the pressure exerted on various objects or various parts of equipment, or on various sections of a fluid system due to a body of fluid in contact with them. Considerations such as the pressure on the bottom of a vertical cylindrical tank, the pressure on the walls and supports of a horizontal cylindrical tank, the height of one fluid necessary to counterbalance a height of another fluid, and instruments for the measurement of pressure are examples of the application of hydrostatics.

The chemical engineer is concerned with hydrodynamics principally to determine power required to move a body of fluid under given conditions, and the effect of moving fluids on rates of other processes taking place in equipment such as the rate of transfer of heat, the rate of mixing, etc.

It is necessary, in a discussion of flow of fluids, to define the various terms used in designating fluid characteristics.

SYMBOLS AND NOMENCLATURE

A = area = square feet
C = constant or coefficient = no units
D = diameter = feet
f = friction factor = no units

F = friction loss corresponding to force in pounds = feet
g = acceleration of gravity = feet per second per second (32.2)
G = mass velocity = $u\rho$ = pounds per square foot per second
H = head or height = feet
K = constant in contraction loss formula = no units
L = length or distance = feet
m = hydraulic radius = cross-section ÷ wetted perimeter = feet
4_m = equivalent diameter = shape factor = feet
P = pressure = pounds per square foot
R = reading = feet
Re = Reynolds number = $\frac{Du\rho}{\mu}$ = no units
u = velocity = feet per second
V = specific volume = $\frac{1}{P}$ = cubic feet per pound, or = flow = cubic feet
W = work added = foot-pounds per pound
z = height, or head = feet
α = angle = degrees
Δ = difference of values = no units
Θ = time = seconds
μ = viscosity = pounds per foot per second
ν = kinematic viscosity = square feet per second
ρ = density = pounds per cubic foot
ϕ = experimental function = no units

Subscripts

A and B refer to materials A and B or to points A and B
c = sudden contraction of cross section
e = sudden enlargement of cross section
f = due to friction
F = due to friction of all sorts including contraction and enlargement
i = due to internal work
o = orifice
P = static pressure
p = pitot
u = velocity
v = venturi
w = work
z = potential

1 and 2 refer to positions 1 and 2 where 1 is upstream from 2

Pressure (P = lbs. per sq. ft.) refers to the total pressure or to the intensity of pressure, and at a particular point is the result of the force of the fluid on that point, and

$$P = \rho H \tag{1}$$

Absolute Pressure is the pressure above that of a perfect vacuum.

Atmospheric Pressure is that due to the atmosphere. Its value, referred to sea level, at 0° C. is 14.595 lbs. per sq. in. = 1 atmosphere.

Gauge Pressure is the pressure in excess of that due to the atmosphere. Gauge pressure plus atmospheric pressure gives absolute pressure.

Impact Pressure is that due to a moving fluid, and is measured in the direction of flow of the fluid.

Static Pressure is the pressure normal to the surface on which it acts. In a moving fluid it is measured at right angles to the direction of flow.

Velocity Pressure is that due to the motion of a fluid and is equal to the impact pressure minus the static pressure.

Head. (H = feet) A head is the height in feet of fluid of uniform density above a reference plane, and rearranging equation (1)

$$H = \frac{P}{\rho} \tag{1a}$$

Atmospheric Pressure Head is that corresponding to atmospheric pressure. 1 atmosphere = 760.0 mm. Hg = 29.92 in. of Hg = 33.9 ft. of H_2O.

Impact Head is that corresponding to impact pressure.

Static Head is that corresponding to static pressure.

Velocity Head is that corresponding to the velocity pressure.

Potential Head is that corresponding to the pressure due to the height of fluid above the reference plane.

Friction Head is that corresponding to the pressure loss due to friction with the walls of a channel.

Contraction Head is that corresponding to the pressure loss due to contraction of cross-section of the channel through which the fluid is flowing.

Enlargement Head is that corresponding to the pressure loss due to enlargement of cross-section of the channel through which the fluid is flowing.

Internal Work Head is that corresponding to the work done on the fluid by internal expansion for one pound of fluid for one second.

Work Head is that corresponding to the external work done on the fluid, or the work added as by a pump, for one pound of fluid for one second.

Lettered subscripts after H refer to the type of head and are indicated in the table of symbols and nomenclature.

Δ refers to a difference and ΔH refers to the decrease of head between points 1 and 2 respectively. To illustrate:

$$\Delta H_u = (H_{u1} - H_{u2}) \tag{2}$$

where the velocity head at point 1 is greater than that at point 2. Bernoulli's Theorem, based on conservation of energy, may be expressed in terms of heads if we consider 1 pound of fluid flowing per second. Referring to Fig. 1, Bernoulli's Theorem applied to the points 1 and 2 states

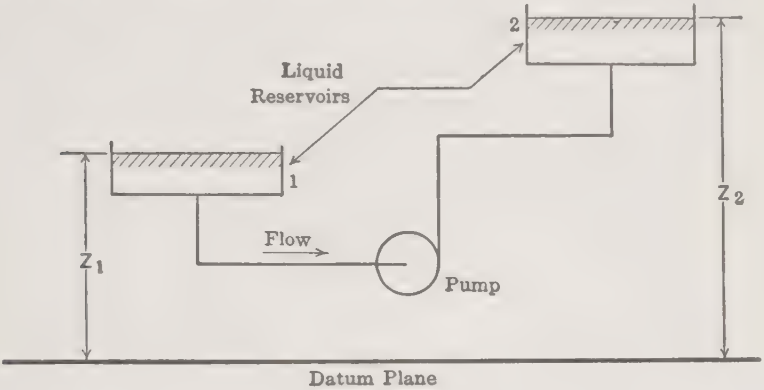


FIG. 1.—Illustration of application of Bernoulli's Theorem.

that the sum of the potential, velocity, static, friction, internal work, and external work heads at point 1 must equal these same heads at point 2. Using symbols, this may be expressed as:

$$H_{z1} + H_{u1} + H_{p1} - \Delta H_F + \Delta H_i + H_w = H_{z2} + H_{u2} + H_{p2} \tag{3}$$

or for external work, on rearranging,

$$H_w = \Delta H_z + \Delta H_u + \Delta H_p + \Delta H_F + \Delta H_i \tag{3a}$$

or as more commonly written,

$$z + \frac{u_1{}^2}{2g} + \frac{P_1}{\rho_1} - F + \int_{v_1}^{v_2} p\,dv + W = z_2 + \frac{u_2{}^2}{2g} + \frac{P_2}{\rho_2} \tag{3b}$$

It should be noted in the preceding equations that:

H_{z1} is the potential head above the datum plane to point 1.

H_{u1} is the velocity head at point 1 and $= \dfrac{u_1{}^2}{2g}$

H_{p1} is the static pressure head at point 1.

H_w is the head corresponding to the work added to the system.

ΔH_F is the head loss due to friction with the walls of the channel plus the loss due to any sudden enlargements or contractions.

ΔH_i is the head loss due to internal energy changes of the fluid; for liquids its value is 0, for gases the term $\int_{v_1}^{v_2} p\,dv$ must be evaluated.

Types of Fluid Flow. Fluid flow may be viscous ("streamline") or turbulent. The type of flow may be determined by a method such as the classical Reynolds experiment, by determining velocity distribution, by photographic or stroboscopic methods, by the Reynolds criterion, or other special means. As a result of Reynolds experiments it has been found that for value of Re less than 2100 for straight pipe, the flow will be viscous and for values of Re over 4000 the flow will be turbulent.

Losses in Head Due to Fluid Flow. When a fluid is flowing through a channel, energy is used up in overcoming friction due to the walls of the channel and the friction resulting from eddy currents due to sudden enlargements and contractions of the cross-section, fittings, etc., through which the fluid is flowing. Such losses in energy are accounted for in terms of heads.

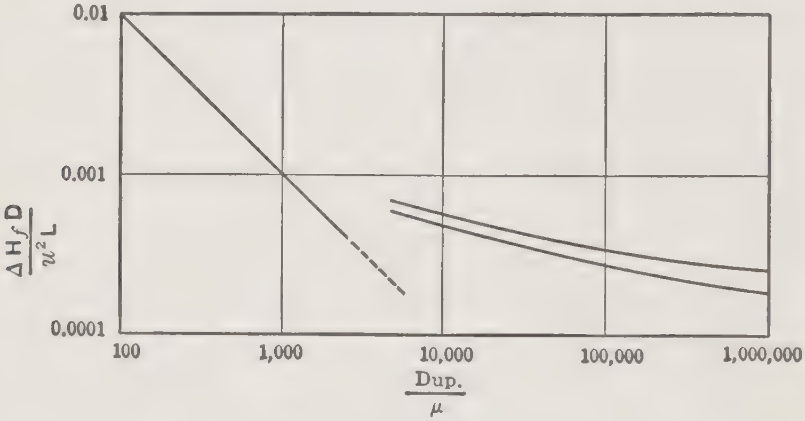


Fig. 2.—Loss in head vs. Reynolds number.

Friction in pipes of constant cross-section may be evaluated by the use of equations based upon the plot in Fig. 2.[1] The equation for viscous flow (Re less than 2100) in terms of head loss is

$$\Delta H_f = \frac{32\mu\rho L u}{g D^2} \tag{4}$$

The equation for turbulent flow (Re greater than 4000) through clean new commercial iron pipe is

$$\frac{\Delta H_f D}{L u^2} = 0.0001906 + \frac{0.01171}{Re^{0.38}} \tag{5}$$

[1] Drew, Koo, and McAdams, Trans. Am. Inst. Chem. Eng., 28, 56 (1932).

and for clean smooth circular pipe (brass, copper or glass) is

$$\frac{\Delta H_f D}{L u^2} = 0.0000870 + \frac{0.00777}{Re^{0.32}} \tag{6}$$

For channels other than circular Fig. 2 and equations (3), (4), and (5) may be used if the term D is replaced by an equivalent diameter. The equivalent diameter ($4m$) is often referred to as the shape factor and is equal to 4 times the hydraulic radius m. The equivalent diameter for an annular space between two concentric circular areas is $4m = D_1 - D_2$, where D_1 and D_2 are the larger and smaller diameters respectively of the two areas.

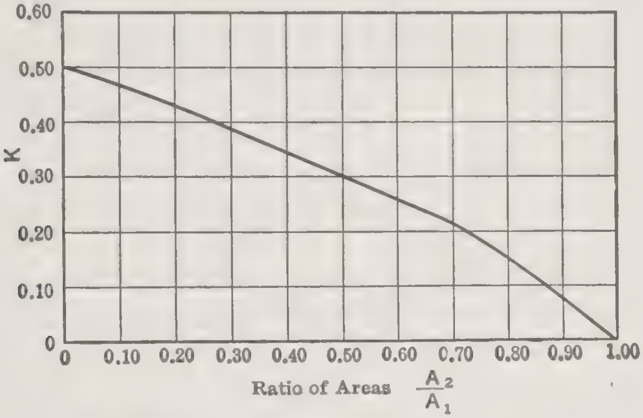


FIG. 3.—Ratio of areas vs. equivalent diameter.

Head losses due to sudden enlargements of cross-section may be evaluated in terms of velocity heads thus:

$$\Delta H_e = \frac{(u_1 - u_2)^2}{2g} \tag{7}$$

where 1 refers to the smaller cross-section and 2 refers to the larger cross-section.

Head losses due to sudden contraction of cross-section may be evaluated in terms of a velocity head, by the use of Fig. 3 and

$$\Delta H_c = \frac{K u_2^2}{2g} \tag{8}$$

where K is determined from Fig. 3, and subscript 2 refers to the smaller cross-section.

Head losses due to pipe fittings may be approximated in terms of equivalent lengths of standard straight pipe of the same size by the use of table 1.

Gases and Vapors. The foregoing relations apply to gases only when the overall pressure drop is not over 10% of the initial pressure. When the pressure drop is greater than this, see *Chemical Engineers' Handbook*, and *Principles of Chemical Engineering*.

MEASURING DEVICES

The basic method used to measure an amount of liquid is by direct weight. To measure a rate of flow the time required for the passage of a definite weight is determined.

The basic method used to measure an amount of gas is by volume (often by displacement of a liquid) or the change of pressure of a reservoir of the gas, and appropriate corrections to standard conditions are necessary. To measure a rate of flow the time required for the passage of a definite amount is determined.

The basic method to measure an amount of a condensible vapor is by weighing the condensate.

Calibration of various metering devices is made by means of such direct weight or volume measurements.

Liquids are often measured by volume or by pressures developed especially when large reservoirs or large enclosed tanks are to be gauged.

Pressures are measured by the barometer or with manometers or Bourdon type gauges.

Fluid flows are conveniently measured by two principal types of equipment: i.e., mechanical meters and hydrodynamic meters.

MECHANICAL METERS

Used for quantity or rate of flow measurement. For a description of operation of various meters consult a standard text or handbook. The meters designed primarily to measure total quantity of fluid which has passed through the meter often may be arranged with appropriate mechanisms to indicate or record rate of flow. The principles used in most meters may be applied to either liquids or gases but it is not often possible to substitute liquid meters for gas meters or vice versa.

Meters for Liquids. The most common types are the disc meter and the current meter. Disc meters are generally constructed with a composition disc which is subject to distortion at temperatures above 125° F. They may be obtained specially with metal discs suitable for higher temperature liquid metering. Their accuracy depends upon clearance of the disc but in general is good except for very low flows. The current meter, or impulse wheel meter, should be used only for high rates of flow. There is too much slippage of liquid to allow measurement of low flows or to give very accurate measurements.

Piston meters are used for accurate measurement of a wide variety of liquids, at various temperatures, various pressures, and various rates, and for intermittent flow.

Meters for Gases. Wet and dry gas meters are common. The wet meters are widely used. They must be maintained level and contain a definite amount of liquid. Generally they are applied to low flows, to non-corrosive gases, and at low pressures.

Dry gas meters are widely used for household gas measurements. They require less attention than the wet meters and are not quite so accurate. They may be used for a rather wide variety of gases and at moderate pressure. They are made in various sizes and large quantities of gas can be measured by them.

For very large flows at low pressures a well-balanced cycloidal blower-type meter can be used. These are common only in industries where large quantities of gas are handled.

The Vane Anemometer when well balanced and calibrated is satisfactory for flows of gas where the area is large, the flow not too low, and for vertical or horizontal flows. Calibration for the particular condition of use is important. The instrument must be handled with extreme care and should never be turned while in a stream of gas. It is best to shield the instrument with a piece of cardboard whenever it is moved through a gas flow of high velocity.

The Cup Anemometer is used only for large flows and is a little more rugged than the vane anemometer.

Addition or Dilution Metering. A number of instruments and methods are available for determining the rate of flow of fluids by determining the rate at which energy or foreign material is absorbed by the fluid or by the instrument. Algebraic relations may be set up between change of conditions and quantity of flow, or electrical instruments may be used to solve such relations automatically and continuously.

Dilution metering, as for example the addition of a measured amount of carbon dioxide to a stream of air and analysis of the air before and after the carbon dioxide addition, is practiced in both the determination of gas and liquid flows. It has a wide range of possibilities and applicability. The chief drawback to its use is the difficulty of obtaining automatic continuous analyses of the stream, and assurance of complete mixing.

The Kata-Thermometer is the simplest thermal instrument in use with small gas flows. Its usefulness depends upon the rate at which heat is removed from a large bulb alcohol thermometer. The instrument is best adapted to low flows of gas and it is the best instrument for velocity of gas currents where the direction of flow is not definitely known. It is conveniently used for gases flowing in any direction but is limited to flows easily accessible.

The Hot-Wire Anemometer is an electrically heated wire and its usefulness depends upon the rate at which heat is removed from the wire, this being a function of the fluid flow. This instrument may be adapted to gases or liquids and should be carefully calibrated. It lends itself to continuous recording.

The Thermal Electric Meters, like the Thomas flow meter for gases, differ from the hot-wire anemometer in that a unit extends over the entire cross-section of a duct and consequently is more accurate. These meters measure the temperature increase of the fluid for a definite amount of heat added between the points where temperatures are taken. They are the same in principle with the dilution method except that heat is added rather than matter. Since the mechanism is electrically activated and analyses are determined electrically, they can be used for continuous recording. These instruments are built for definite installations for definite size ducts and are not readily adapted to changes of location or service.

HYDROSTATIC INSTRUMENTS

Measurement of Pressures. Pressures may be measured by means of pressure gauges or manometers. *Mechanical pressure gauges* of the Bourdon type are the most common industrially. Such gauges are available in a variety of sizes, ranges, and materials of construction. They are made for pressures from zero pounds per square inch absolute to thousands of pounds per square inch. They are accurate only after calibration and if handled with care. Calibration is made with a dead-weight gauge tester for pressures above atmospheric, and with a mercury manometer for pressures lower than atmospheric. It is important to make a calibration over the whole range of the instrument if accurate results are desired. It should be protected from violent fluctuations, undue temperature changes, and should be properly installed. Care must be exercised so that the pipe connecting the gauge to the point of measurement does not become liquid or gas bound and thereby cause a false reading.

Manometers. All discussion of manometers is based upon those where visual readings may be made of fluids inside glass tubing, of such size that the capillary effect can be neglected. The simplest manometer consists of an open tube of glass connected to a body of fluid and showing by the height of that fluid the pressure at the point of connection. Such a tube is often referred to as a piezometer tube. Open piezometer tubes are generally used only in work with water, or as a means of gauging the contents of a tank or reservoir. The pressure above atmospheric may be computed directly from equation (1) where the reading R is equal to the head H. See Fig. 4a.

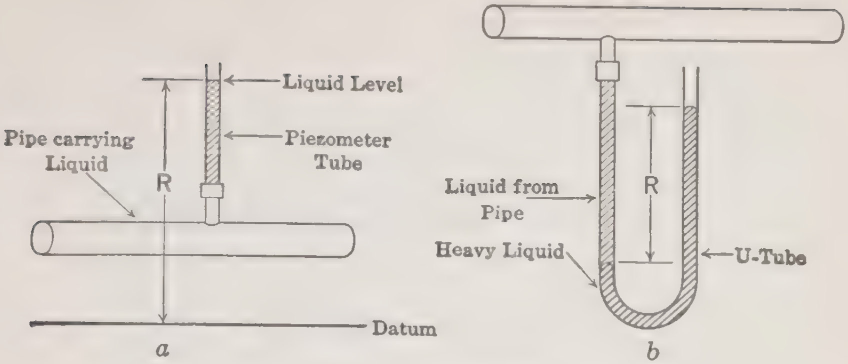


FIG. 4.—Manometer.

The most common form of manometer is a modification of a U-tube where the indicating liquid differs from the fluid whose pressure is desired. See Fig. 4b. Here the manometer reads a pressure difference between points 1 and 2, and it is seen that the reading R, while showing a height of mercury, does not equal the pressure difference computed by equation (1) where the density of the fluid measuring the head H is required. Thus, if the head used in equation (1) is R, the density becomes the difference in densities of the two fluids, or

$$P = P_1 - P_2 = R(\rho_A - \rho_B) \qquad (9)$$

When the densities of the two fluids A and B are quite different, as for example when A is water and B is air at 60° F., saturated, the difference between ρ_A and ρ_B is only slightly less than ρ_A so that the density of A may be used directly. In this case an error of less than 0.12% would result. If, however, A is mercury and B is a brine of sp. gr. = 1.25, the difference in densities must be taken into account, for to use the density of mercury alone would result in an error of over 10%.

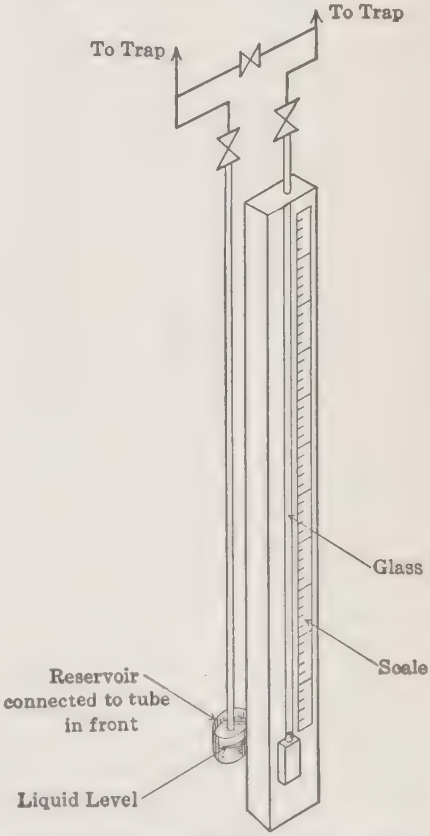
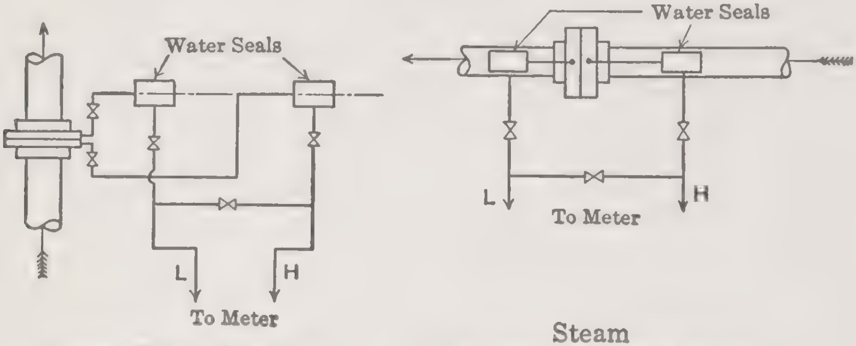


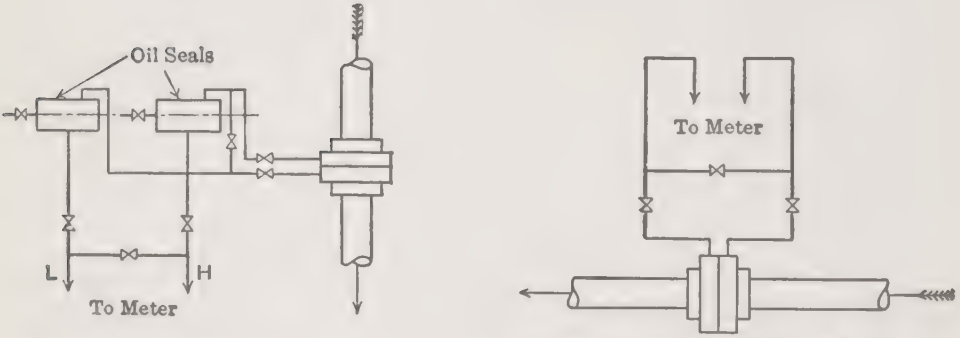
FIG. 5.—Modification of customary U-tube.

Figure 5 shows one modificaton of the simple U-tube of Fig. 4b such as is commonly found in chemical engineering work. The principal details of construction are given and the usual connecting lines, traps, and valves are shown. It is advisable to have the scale ruled on brass or steel, and if very precise pressures are desired it is necessary to correct all readings to account for the difference in expansion of the manometer fluid and the scale, as is done for the barometer. The traps may be used for a two-fold purpose, both to save the manometer fluid if it is blown out of the manometer, and also to act as a seal if the instrument is to be used in connection with a condensible vapor. For example, if the manometer is connected to a steam line so that steam fills the lines *C* and *D*, the tubes over the mercury should be free from water which will condense in the lines but will be kept out by the traps. As shown in Fig. 6 such traps may more properly be placed near the line containing the fluid. Manometers of this general type are commonly used where line pressures may be as high as 150 lbs. per sq. in. gauge. In Fig. 5 are shown the desired



Steam, Vertical upflow

Steam
Horizontal, above meter



Heavy Oil Vertical downflow

Water, Light Oil, Gas & Air
Horizontal flow below meter

Fig. 6

valve connections for a manometer. It is customary to place a small plug cock valve (C) in a by-pass line connecting the two pressure leads. Between readings of the manometer this valve should be kept open and should be kept closed only when readings are being taken. The use of this valve is a safety precaution and prevents loss of manometer fluid either into the lines or into the traps when there are large pressure differences being measured and where there may be wide fluctuations in pressure.

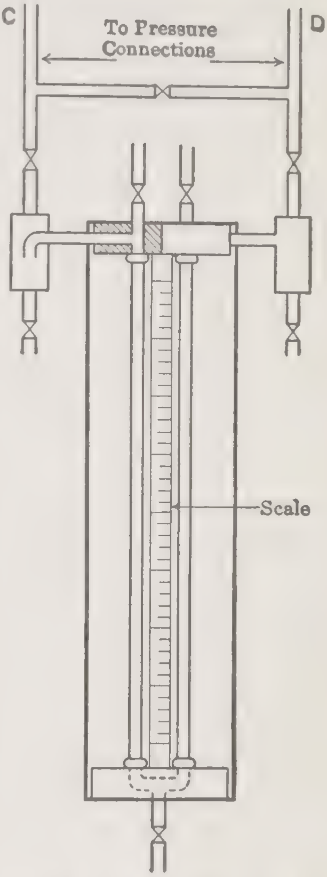


Fig. 7.—Modification of U-tube manometer.

A modification of the U-tube manometer is the well type, a sketch of which is shown in Fig. 7. In this manometer only one level is read, whereas in the U-tube manometer the level in both tubes must be read.

Another modification of the simple manometer is the inclined tube manometer or draft gauge. The inclined U-tubes are used for very low pressure differences for both liquids and gases. The draft gauge is used for low pressure differences of gases only. A diagram of the draft gauge is shown in Fig. 8. This figure also shows the valves between the gauge and the point of pressure reading. These valves on the draft gauge are three-way valves and may be set to operate so that either end of the instrument may be opened to the atmosphere or to the pressure point. By manipulation of valve A and B so that they are open in the position shown, the reading on the draft gauge will indicate the difference between the high pressure and low pressure. Then, by turning valve B so that atmospheric pressure is

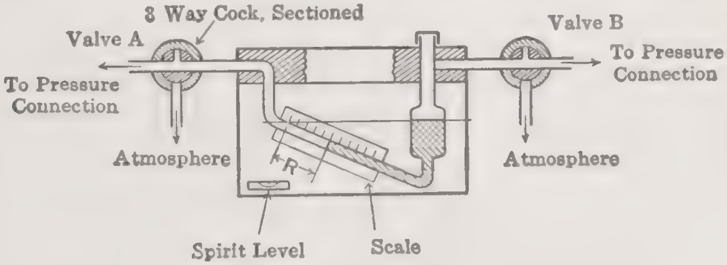


Fig. 8.—Inclined tube manometer or draft gauge.

on that side of the instrument, the reading will be equal to the high pressure. From these two readings the value of the low pressure can be determined.

Two-fluid U-tube manometers are often used for measuring very low pressure difference. See Fig. 9. In this instrument the magnitude of the reading *R* is dependent upon the difference in density of the two fluids used. The smaller this difference in density, the larger will be the reading *R* for the same pressure difference. Since the reading is very sensitive to a small change in density, it is important that the exact density of the two fluids used be known accurately at the temperature of operation. Often in this type of manometer fluids are used which have a very low solubility in each other, and it is important that the two resulting solutions be in equilibrium with each other at the temperature of operation. The safest and most direct method for obtaining accurate results with this sensitive instrument is to calibrate the instrument at frequent intervels with a U-tube single liquid manometer connected as shown in Fig. 9. This manometer must contain a liquid of known density. A few readings made with the calibration manometer are sufficient to determine the ratio between the density of the single liquid to the difference in densities of the liquids in the two-liquid manometer.

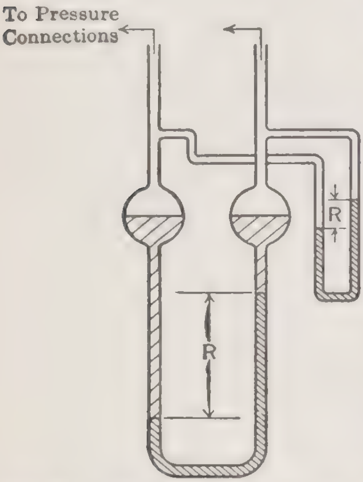


FIG. 9.—A two fluid, U-tube manometer.

HYDRODYNAMIC MEASURING METHODS

These methods are all dependent upon the relation between a difference in pressure at two points in a moving fluid and the relation between such pressures and the rate of flow. Four types of measuring devices may be classed under this heading. They are Pitot tubes, Venturi meters, orifices, and weirs. The first three are used for measuring both liquids and gases, while weirs are used for the measurement of liquids only. The Pitot tube, the Venturi meter, and the orifice are all used in conjunction with an appropriate manometer, the choice of the manometer depending upon the magnitude of the pressure difference to be determined. The weir requires only the use of a Hook gauge or some other level height determining instrument.

Pitot Tube. A Pitot tube with recommended dimensions[1] is shown in Fig. 10. The Pitot tube has a connection *A* by which impact pressure may be transferred through opening A', and a connection B by which static pressure may be transferred through the static openings B'. The instrument is used in either liquid or gas streams but its greatest use is in the latter. It is essential that the impact opening be pointed in the exact direction of the flowing fluid at the point of measurement. The tube may be pointed upstream or downstream but it is customary to use it pointing upstream.

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[1] A.S.M.E. Research Publication, "Fluid Meters." 3d Ed., 1931, p. 43.

The two connections A and B of the Pitot tube are connected to the two sides of a manometer. In the case of gases the manometer is of the draft gauge type. By appropriate manipulation of the valves A and B on the draft gauge in Fig. 8, the difference between impact pressure head and static pressure head is found, and also when B is opened to the atmosphere the static pressure head is read. From this the impact head can be calculated. The difference between the impact head and the static head, or the first head read, is the velocity head. The velocity head is related to the actual velocity by

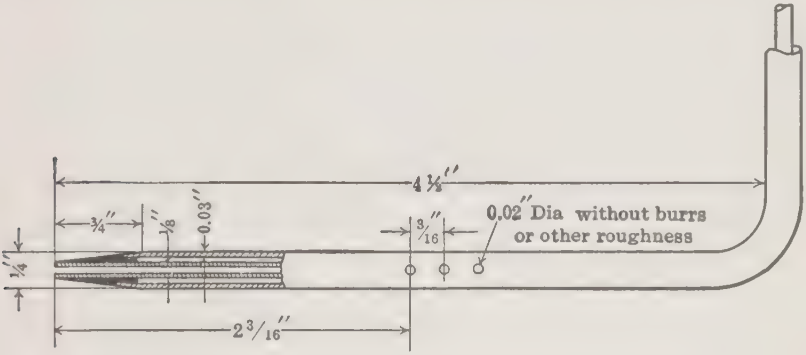

FIG. 10.—Pitot tube with recommended dimensions.

$$\Delta H = \frac{u^2}{2g} \tag{10}$$

If there is no turbulence caused by the Pitot tube, and if the openings are sufficiently small, the velocity may be calculated by the equation

$$u = \sqrt{2g\Delta H} \tag{10a}$$

Actually, while no Pitot tube can be perfectly stream-lined, it is common to find a well-made Pitot tube which has a coefficient of 0.98 to 0.99, where the coefficient is C_p in the equation

$$u = C_p\sqrt{2g\Delta H} \tag{10b}$$

To measure the velocity of flow in a pipe or duct by means of a Pitot tube, it is necessary that the pipe be "explored" and that one or more traverses of the channel be made. Due to swirls and eddy currents which may be present in the body of a flowing fluid, it is often necessary to make traverses in different planes and along different pipe diameters. In the case of a square or rectangular duct, the cross-sectional area of the duct should be divided into a convenient number of equal area squares or rectangles. Then by placing the Pitot tube at the center of these areas a series of readings are made. From these readings the velocity at each point can be calculated, and the average of these velocities is the average velocity in the duct.

In the case of a round duct with fluid flowing, the cross-sectional area of the pipe is divided into a number of equal area concentric rings. Pitot tube readings are made along a diameter at points where that diameter intersects circles which bisect the equal area annuli. In order to obtain an even number of readings the central area, which is circular, is divided by a circle so that it is bisected, and the two points where the diameter crosses this circle are taken rather than the center point of the central area. From these readings of velocity head the velocity at each point can be calculated, and the average of these velocities is the average velocity across the particular diameter traversed. Usually, either a horizontal or vertical diameter is used. For an accurate exploration of a pipe, both vertical and horizontal explorations are necessary, and often exploration at several other diameters is required. For a good degree of accuracy it is customary to make a ten point traverse. In addition to the traverse point readings a center point reading should be made. The relation between the center point velocity and the average velocity can be determined, and after such a calibration the velocity of flow in any type of duct can be determined by means of a center velocity reading only.

Example 1: Air is being blown through a 12″ diameter pipe. An eight point traverse has been made with a Pitot tube along the horizontal diameter to determine the rate of air flow and the power required by the blower. From the following test data find:

(a) The velocity at different points across the diameter, and plot the distribution.
(b) Flow of air in cubic feet per minute (C.F.M.).
(c) Horsepower required at the blower, neglecting friction.

Dry bulb temperature of air 60° F.
Wet bulb temperature of air 48° F.
Atmospheric pressure 29.92″ Hg.

Traverse Point	Velocity Head, Inches of Water	Impact Head, Inches of Water
1	0.60	1.10
2	1.20	1.71
3	1.25	1.76
4	1.35	1.86
5	1.40	1.91
6	1.35	1.85
7	1.30	1.80
8	1.10	1.61
Center	1.35	1.86

Solution. (a) To locate the traverse points, divide the pipe into four equal concentric areas and then divide each of these into two equal areas, taking readings at the intersection of these latter bisectors with a diameter.

Let $a_1 = a_2 = a_3 = a_4$, and r_1 = radius from center of pipe for the circle bisecting a_1.

$$a_1 + a_2 + a_3 + a_4 = A = \pi(6)^2 \text{ sq. in.},$$

since $$\frac{a_1}{2} = \pi r^2, \text{ and } a = \frac{A}{4}$$

then $$r_1 = \sqrt{\frac{(a \div 2)}{\pi}} = 2.12'' \qquad r_3 = \sqrt{\frac{(a \div 2) + 2a}{\pi}} = 4.74''$$

$$r_2 = \sqrt{\frac{(a \div 2) + a}{\pi}} = 3.67'' \qquad r_4 = \sqrt{\frac{(a \div 2) + 3a}{\pi}} = 5.61''$$

At these conditions $\rho_{\text{air}} = 0.0757$; then 1 foot of water $= \frac{62.4}{0.0757}$ $= 825.0$ ft. of air. Thus for air velocity $u^2 = \frac{2g(825)\Delta H}{12} = 4430\ \Delta H$ where ΔH = inches of water.

Traverse Point	Location from Side of Pipe, Inches	Velocity Head, Inches of Water	u^2	Velocity, Ft./Sec. u	Static Head, Inches of Water
1	0.39	0.60	2660	51.6	0.50
2	1.26	1.20	5320	72.9	0.51
3	2.33	1.25	5540	74.5	0.51
4	3.88	1.35	5980	77.4	0.51
5	8.12	1.40	6200	78.7	0.51
6	9.67	1.35	5980	77.4	0.50
7	10.74	1.30	5760	75.9	0.50
8	11.61	1.10	4870	69.8	0.51
			Ave.	= 72.3	0.506
Center......	6.00	1.35	5980	77.4	

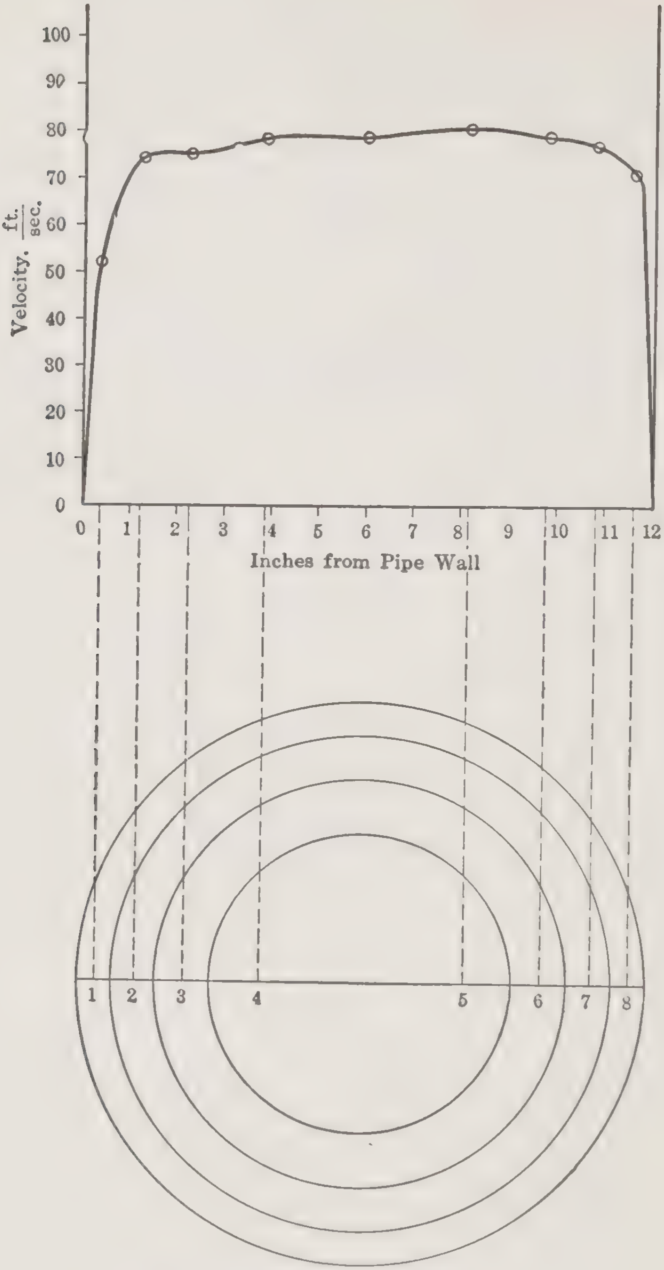

Fig. 11.—Velocity distribution.

Velocity distribution plot is given in Fig. 11.

(*b*) $\text{velocity} = \dfrac{72.3\pi(1)^2 60}{4} = 3410 \text{ C.F.M.}$

(*c*) $\text{static head} = 0.506\left(\dfrac{825}{12}\right) = 34.8 \text{ ft. of air}$

$\text{velocity head} = \dfrac{u^2}{2g} = \dfrac{(72.3)^2}{64.4} = 81.3 \text{ ft. of air}$

total head at plane of traverse = 34.8 + 81.3 = 116.1 ft. of air

$\text{Air h.p.} = \dfrac{116.1(0.0757)3410}{33{,}000} = 0.908 \text{ h.p.}$

Venturi Meter. Fig. 12 shows a Venturi meter and the points of connection for a manometer. As fluid travels from *A* to *B* the velocity increases. The velocity head at *B* is greater than the velocity head at *A* at the expense of the static head at *A*. The static pressure connection at the throat *B* will indicate a lower static pressure than the static pressure at point *A*. And if all this static pressure head is converted to velocity head, it follows that the difference in velocity head will be

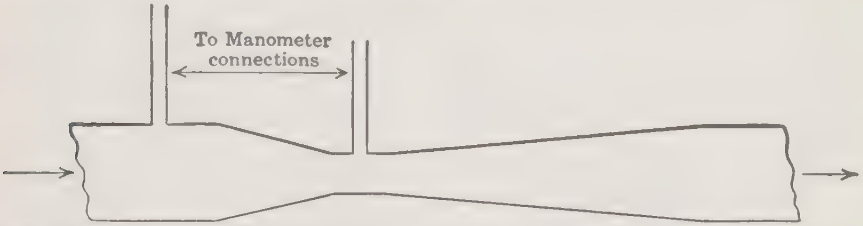

FIG. 12.—Venturi meter, showing points of connection for manometer.

$$\Delta H = \frac{u_B{}^2 - u_A{}^2}{2g} \tag{12}$$

Venturi meters are built so that there is the minimum of friction and eddy current loss when fluid is flowing through the instrument. However, there is a slight amount of friction and turbulence through any Venturi meter, and for this reason not all the static head loss from *A* to *B* is converted to velocity head gain. By the use of the equation:

$$\Delta H = \frac{u_B{}^2 - u_A{}^2}{C_v{}^2 2g} \quad \text{or} \quad \sqrt{u_B{}^2 - u_A{}^2} = C_v\sqrt{2g\Delta H} \tag{13}$$

the relation between velocity and static head loss can be determined. C_v is the Venturi meter coefficient. For a well built Venturi meter it may be equal to 0.98. There is very little permanent head loss for fluids flowing through Venturi meters. See *Chemical Engineers' Handbook* for dimensions and head losses for a Venturi meter.

This instrument therefore can be inserted in a pipe line for the measurement of a flow and require only a small power consumption. The manometer attached to the meter should be chosen according to the pressure differences to be measured.

Orifices. An orifice is shown with manometer connections in Fig. 13. Several types of orifice plates are used for this type of meter. The sharp edge orifice is the most common, while the round edge orifice causes less permanent head loss. Connections of the manometer should be made at points on the upstream and downstream sides of the orifice, such that the maximum difference in head can be measured. These points are dependent on the size of the pipe and the size of the orifice.[1]

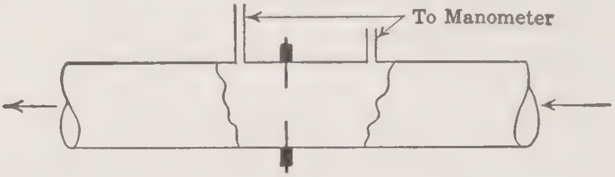

FIG. 13.—Orifice plate, showing manometer connection.

As in the Venturi meter, pressure head on the upstream side of the orifice is transferred to velocity head through the orifice. Due to the sudden contraction and enlargement of the stream of fluid there is considerable head loss in friction and the formation of eddy currents, with the result that considerable energy is used up when a fluid flows through an orifice meter.

The relation between the flow through an orifice and the difference in head across the orifice is given by the following equation:

$$\Delta H = \frac{u_0^2 - u_A^2}{C_0^2 2g} \quad \text{or} \quad \sqrt{u_0^2 - u_A^2} = C_0\sqrt{2g\Delta H} \tag{14}$$

where C_0 is the coefficient of discharge of the orifice and its value may be determined by the size of pipe, the size of the orifice, and the points at which pressure readings are taken. When the ratio of diameter of orifice to diameter of pipe is small, u_A^2 may be neglected. Fig. 14 shows how various types of orifices may be placed in a meter. It should be noted that most orifice installations do not conform to exact recommendations for size and points of pressure reading. Orifices may be installed in wide varieties of sizes and may have pressure connections at practically any point. When such is the case, it is imperative that the orifice be calibrated in position.

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[1] Badger and McCabe, "Elements of Chemical Engineering."

Example 2. An oil having a sp. gr. of 0.78 is flowing through an orifice whose diameter is 1.5″. The orifice is placed between flanges in a 6″ standard iron pipe. Pressure taps are located 5″ from the orifice on both the upstream and downstream sides. The taps are connected to a mercury manometer. Oil fills the manometer above the mercury and the reading is 12.5″. What is the rate of flow of oil in gallons per minute and what is the horsepower required to force the oil through the orifice?

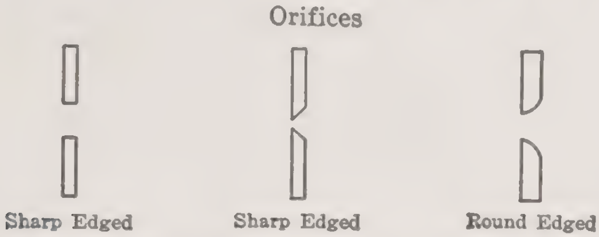

Fig. 14.—Illustration of various types of orifices.

Solution. Ratio of orifice diameter to inside pipe diameter $= \frac{1.5}{6.065} = 0.247$. Refer to Fig. 18, p. 47, *Elements of Chemical Engineering*, second edition. From this figure it is seen that the downstream pressure connection 5″ from the orifice is between the maximum and minimum distance for use of the two lower plots.

From the orifice coefficient plot, $C_0 = 0.601$.

Since the ratio of diameters is small, $\sqrt{u_0^2 - u_A^2}$ is very nearly equal to u_0 and equation (13) becomes $u_0 = C_0\sqrt{2g\Delta H}$;

then $$\Delta H_{\text{oil}} = \frac{12.5}{12}\left|\frac{(13.6 - 0.78)}{0.78}\right. = 17.15 \text{ ft. of oil}$$

and $$u_0 = 0.601\sqrt{64.4(17.15)} = 19.95 \text{ ft. per sec.}$$

$C - ft^3 \times 7.48$

and flow $$= \frac{19.95|\pi|(1.5)^2|7.48|60}{|4|\;144\;|\quad|} = 110.0 \text{ gal. per min.}$$

From the plot the permanent head loss through the orifice is 86.5% of the pressure drop across the orifice; then

$$\frac{17.15|62.4|0.78|\;110|\;0.865}{|\quad|\quad|7.48|33{,}000} = 0.321 \text{ h.p. required for the orifice.}$$

When orifices are used to measure gases such as air or steam, equation (14) will apply only when there is a very small pressure drop across the orifice. It is generally necessary to calibrate orifices for such use. In some cases, and within definite limitations, equations such as those in the texts[1] can be used.

Weirs. Weirs are generally limited to the measurement of flow of liquids. Weirs may be made with either rectangular, V-notch, or slot openings, depending upon the amount of liquid being measured. The head on the weir is the distance between the lowest edge of the notch and the height of the liquid above the notch at a point of maximum liquid height. Unless rather elaborate precautions are taken to insure a very steady and non-turbulent flow at the point where the level is determined, it is necessary to calibrate the weir. There are a number of formulas applying to various types of weirs, which can be found in the handbooks.[1] The best approximation for an equation to be used with a sixty degree V-notch weir is:

$$\frac{V}{\theta} = 2.505 \left(\tan \frac{\alpha}{2}\right)^{0.996} H^{2.47} \tag{15}$$

where V is the flow in cubic feet.

Weirs cannot be used in a closed pipe system unless some provision is made whereby a glass panel can be used so that the height of liquid above the weir may be observed. Many "tail-boxes" are made up in the form of weirs so that products flowing from a reaction vessel may be observed and their quantities estimated.

PUMPS AND BLOWERS

Liquids are pumped in a variety of ways. The air lift is sometimes used in the chemical industries but liquids are generally moved by reciprocating pumps, rotary pumps, or centrifugal pumps. Consult handbooks and catalogues for various pump characteristics. A few important notes concerning pump performance and use are:

Reciprocating pumps give pulsating flows. They will handle liquids over wide ranges of temperature and can produce very large heads. Reciprocating pumps are controlled by the length and speed of the stroke, and are simple to control and to maintain.

Rotary pumps can be used for nearly all kinds of liquids and will produce reasonably high heads. Rotary pumps are generally operated at constant speed and have a by-pass which can be regulated to control the output of the pump.

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[1] Walker, Lewis, McAdams, and Gilliland, "Principles of Chemical Engineering"; Perry, "Chemical Engineers' Handbook."

Centrifugal pumps can be used for any type of liquid and are successfully used for liquids containing suspended particles. They are easy to maintain and will produce high heads if properly powered. Centrifugal pumps are generally connected to constant speed motors and their output controlled by a valve on the discharge side of the pump.

Pump efficiencies are determined by dividing the total work done by the pump on the liquid by the total power input to the pump. When an overall efficiency of pump and motor is referred to, this efficiency means the ratio of work done on the liquid divided by power input to the motor.

EXPERIMENT NO. 6

Viscous and Turbulent Flow of Water

Object. 1. To observe visually the difference between stream-line and turbulent flow.

2. To observe the rapid transition from one type of flow to the other.

3. To determine quantitatively the transition point between stream-line and turbulent flow by means of Reynolds number.

THEORY

Reynolds found that when a colored fluid was introduced into a stream of slowly moving water the stream of colored fluid would remain intact as a separate stream if the rate of flow of the water was sufficiently low; such flow is called viscous flow. Above a certain rate of flow of the water the stream of colored fluid would mix rapidly throughout the main body of the fluid, and this type of flow is called turbulent. The state of motion (viscous or turbulent) has been found to be a function of the size and shape of the channel, the velocity of flow, the density of the fluid, and the viscosity of the fluid. For any fluid flowing through a channel there is a critical value of the so-called Reynolds number below which the flow is said to be viscous and above which the flow is said to be turbulent.

Equipment. Glass apparatus as in Fig. 15 will show both qualitatively and quantitatively the difference between viscous and turbulent flow of water. Rubber tubing is used to connect the water supply and the colored fluid reservoir to the apparatus. A small globe valve may be used to adjust the water supply. Screw clamps permit easy and small adjustments to be made of the colored fluid stream. To determine quantitatively the rate of flow of water, the discharge can be made into a bucket or tank placed upon scales, and the weight of water discharged over a measured length of time determined.

Procedure. Water is allowed to fill the equipment and to flow into the weighing tank. After the equipment is filled, the level of the reservoir of colored fluid can be fixed and its flow adjusted so that a small stream is allowed to flow from the tip of the jet into the main body of the water. Adjustment should be made so that the velocity of the colored fluid will be approximately that of the water. A solution of $KMnO_4$ in water can be used as the colored fluid. Other substances may be used if desired but the color of $KMnO_4$ is intense and has proven to be satisfactory for this experiment.

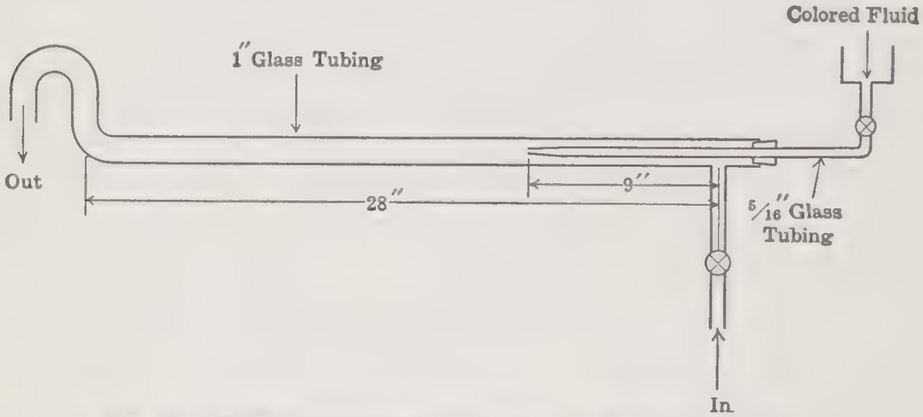


FIG. 15.—Glass apparatus for illustrating Reynolds number.

Start a flow of water, introduce the permanganate solution, and adjust the water flow until viscous flow is indicated by the stream of permanganate solution remaining unbroken throughout the tube. Increase the flow of water until turbulent flow is indicated by the stream of solution being completely disrupted soon after issuing from the jet.

To determine the velocity of flow below which the flow is viscous, or above which the flow is turbulent, perform at least four experiments. In two experiments operate so that viscous flow is observed and increase the rate of water until it seems by observation that the flow is about to become turbulent. When this point has been reached, allow at least 25 pounds of water to pass through the apparatus and find the time required for its passage. In the other two experiments start with turbulent flow and decrease the flow until it is thought that any further decrease will produce viscous flow. At this point take the time required to discharge a given weight of water. Record the temperature of the water.

REPORT

1. Account for any unlooked-for or unusual appearance of the colored fluid stream in both viscous and turbulent flow.
2. Does the transition between viscous and turbulent flow appear to be very critical? Why is this so?

3. From data obtained in the experiments, calculate the Reynolds number for each rate of flow used and from these values determine the critical Reynolds number. Compare this Reynolds number with that given in the texts. Is there any reason for a discrepancy between these values?

EXPERIMENT NO. 7

Calibration of an Orifice and Other Flow-Measuring Instruments

Object. 1. To operate manometers, orifices, and other measuring instruments.

2. To calibrate various flow-measuring instruments.

3. To compare different size orifices.

4. To compare different flow-measuring instruments.

5. To observe the effect of turbulence on the length of calming section necessary for reliable static pressure readings.

THEORY

Metering instruments are calibrated by direct weighing of the liquid flowing through the equipment. To make reliable static pressure observations it is essential that there be no turbulence at the point of reading, due to pipe fittings, projections, etc. For this reason sufficient calming section should be allowed after any bend or projection into a pipe which would cause undue turbulence at the point of static pressure connection.

Equipment. Equipment arranged as shown in Fig. 16 is used. It may be modified to include any other type of measuring instruments. When water is being used at room temperature, the water main may be connected directly to the system and the water discharged from the weight tank into the drain. If it is desired to use water at any other temperature, or to use any other fluid, such as a brine or an oil, the tank *A* can be used as a reservoir and the pump used to cause flow through the system. If a fluid is to be used at a given high or low temperature, it is important that the fluid be recirculated and that the pipes be insulated so that steady conditions can be obtained. After weighing, the fluid can be pumped into tank *A* and another run made. If it is desired to control the temperature, the fluid may be pumped through a heat exchanger located anywhere that is convenient between the pump and ½ inch pipe. The static pressure tubes attached to the 20 foot length of pipe, marked *C*, *D*, *E*, and *F*, must be carefully connected in such a way that there will be a minimum of turbulence in the liquid due to them. It is imperative that points *C*, *D*, *E*, and *F* be at exactly the same level. The orifice may be made in any convenient form and should not necessarily conform to standard orifice practice, i.e., the upstream and downstream pressure connections should be made at any convenient position, but if desired these connections may be made at the proper points to insure an orifice coefficient of 0.61 if the proper size orifice is used. Several orifice plates should be used in different experiments, each having a different size opening. It is also desirable to have one orifice with one edge rounded. These orifices may then be compared in performance. The rotameter shown in the figure may be made up or obtained from a manufacturer. It will not only give a reading of the rate of flow but will also show the steadiness of the flow conditions. The weir may be of any desired type; however, it has been found satisfactory to use a slot type with the slot ¼ inch wide. For the set-up as indicated and using the test section made of ½ inch pipe, a slot 9 inches high will be ample for most conditions of operation. A slot of any other size can be put into a pipe or plate and this plate substituted to give a different range for the weir. The manometer on the orifice is of the usual mercury type. The manometer measuring the head loss due to friction can be of several types, preferably a two-fluid differential manometer made up with some fluid im-

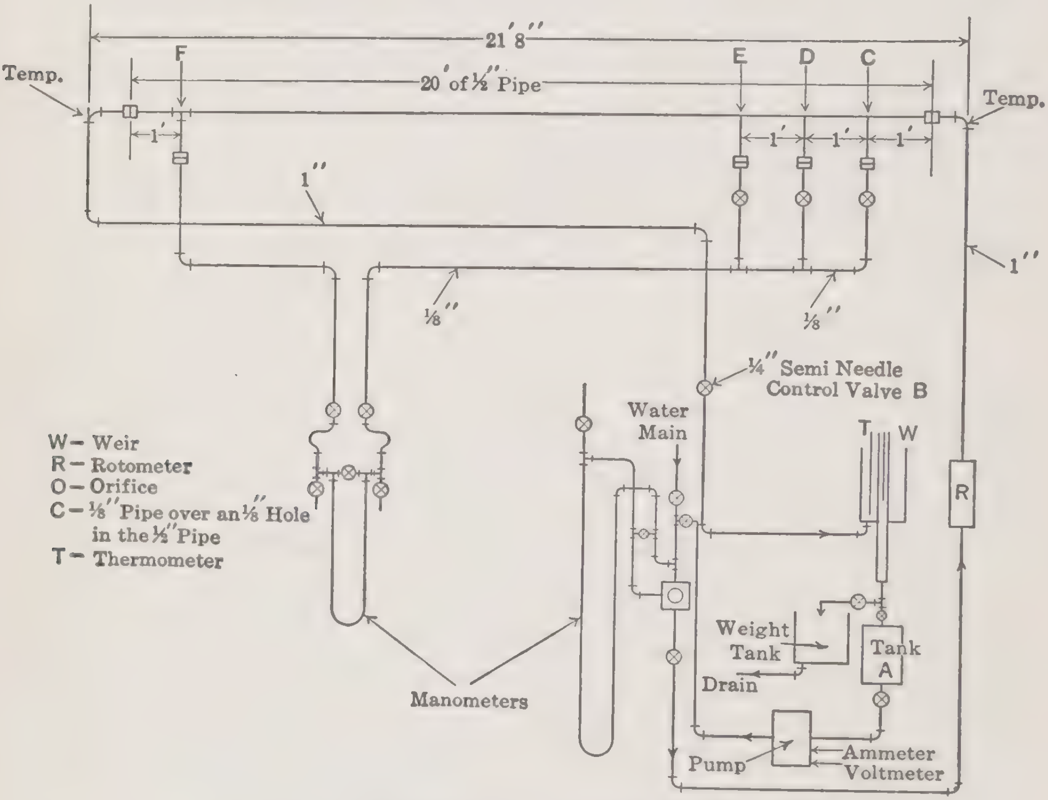


FIG. 16.—Equipment for studying fluid flow.

miscible with the fluid flowing and having but a slight difference in density.

Procedure. See that all manometers are completely and properly filled with liquids and that their zero reading is correct. Allow water to flow through the system, controlling the flow through valve *B* (unless a rotary pump is being used, in which case control with by-pass valve). Since the head and the liquid flowing over the weir are visible, it is easy to determine when conditions of flow are steady. Make all determinations with at least 200 pounds of water and make check runs for each rate of flow used. Record water temperature. For the first determination use the highest rate of flow permissible with the orifice manometer being used. Make five other runs at rates such that the manometer reading will be reduced by approximately even amounts. Make readings on each measuring instrument. Results should be plotted after each determination. Make these laboratory plots by plotting the head observed across the instrument against the time required for the discharge for the given weight of water. Plot these results as soon as they are obtained. A curve can be drawn during the progress of the experiment. If any points are obtained which do not fall on a smooth curve when compared with the other points, check the results immediately by a duplicate run. The highest rate of flow run should be made first so that the limits for the coordinates for the laboratory plot can be determined. Succeeding runs at lower flows are then made. Repeat, using a different orifice plate and a different weir.

To determine the length of calming section necessary to give reliable static pressure readings, open the valves on manometer *B* so that a pressure difference may be read between point *F* and point *C*, *D*, or *E*. First determine the pressure drop at a high rate of flow between *E* and *F*, then the pressure drop between *D* and *F*, and finally the pressure drop between *C* and *F*. Calculate the pressure drop per foot of pipe from the reading between *E* and *F*, and, using this as a basis, compare the pressure drop between *C*, *D*, and *E* and *F*. Repeat for different rates of flow.

REPORT

1. Draw calibration curves showing discharge through the metering devices in gal. per min. versus manometer or head reading.
2. Plot head observed versus rates of flow in both ft. per sec. and gal. per min. on log-log paper.
3. Do these results confirm the general equation for flow through an orifice and a weir? Derive empirical equations for the particular orifices and weirs used and evaluate any coefficients and constants which may appear in these equations. Compare the coefficients with those given in the texts for the orifice sizes and weirs used.

4. Compare the accuracy and range of optimum usefulness of all the orifices and other meters tested.
5. Compare the pressure drop between the points *C-F*, *D-F*, and *E-F*, and determine whether the turbulence caused by the fittings on the upstream side of point *C* affects the static pressure readings at points *C*, *D*, and *E*.
6. How much calming section is necessary for various rates of fluid flow?

EXPERIMENT NO. 8

FLUID FLOW IN A PIPE

Friction Head Loss for Viscous and Turbulent Flow

Object. 1. To determine, for water or other fluids flowing through standard size piping, the loss in fluid head due to friction. The effect of different velocities and different temperatures are to be found.

2. To determine the Reynolds number for various flows.

3. To compare experimental results with values of head losses calculated from the Fanning equation.

4. To determine a critical Reynolds number for a given fluid and temperature.

THEORY

From the Fanning equation it is possible to determine the head loss due to friction of a fluid flowing through a pipe if the size and shape of the channel, the velocity of flow, the density of the fluid, and the viscosity of the fluid are known. It is convenient to use the plot, Fig. 2, for this relationship. It has been found that the head loss due to friction is a function of the Reynolds number. It is also known that the head loss due to friction is a function of the type of flow, i.e., whether viscous or turbulent. The head loss due to friction can be measured by static pressure readings and these losses can then be compared with those calculated by the corresponding Reynolds number and the Fanning equation. From the Reynolds number flows can be determined to be viscous or turbulent and the head losses due to friction can be determined for both types of flow.

Equipment. Equipment arranged as shown in Fig. 16 is used. The measuring devices should have been calibrated previously in Experiment 7. For high or low temperature experiments connect a heat exchanger as suggested in Experiment 7.

Procedure. Operate with water direct from the main, or with other liquids by recirculation directly from the weir to tank *A* to the pump and through the system. Control the flow by valve *B* or a by-pass valve around the pump. Make at least eight runs, varying the velocity of flow in the test section (½ inch pipe) from 0.75 to 0.10 feet per second. Take readings of the pressure drop in the ½ inch pipe for each run. Use points *C* and *F* or *D* and *F*, or *E* and *F*, depending upon which set has previously been decided upon as most dependable from Experiment 2. Record temperatures. Use either the orifice readings or weir readings to determine steadiness of operation and rate of flow. Make at least three readings of all instruments at five minute intervals after steady conditions are reached, and average these for final values.

Repeat, using one or more water temperatures.

Repeat, using a solution or a brine.

If experiments are run with oil, it is important that the equipment be cleaned as thoroughly as possible before being used again for water. It is recommended that steam be blown through the lines for a long time and at a high velocity in order to clean the apparatus.

REPORT

1. Plot friction head versus velocity of flow.
2. Calculate Reynolds number for each experiment and plot these values versus $\frac{\Delta H_f D}{u^2 L}$ on log-log paper, using the observed values of ΔH_f. Compare this plot with the Fanning equation plot in Fig. 2.
3. Using the calculated values of Re determine ΔH_f from Fig. 2, and compare with the observed values of ΔH_f.
4. From the plot (Part 2) of experimental data determine the critical value of Reynolds number. Does this agree with the usually accepted critical value? Explain. What is the critical velocity in the ½ inch pipe?

EXPERIMENT NO. 9

FLOW OF WATER THROUGH AN ORIFICE

Object. 1. To observe visually the lines of flow of a liquid at and near an orifice.

2. To observe visually and to measure the static pressures at various points near the orifice.

3. To calibrate the orifice and determine coefficients of an equation for use with it.

THEORY

With a glass apparatus and using water with some colored or suspended material in it, it is possible to observe the lines of flow of water through an orifice. Piezometer tubes connected to give static pressure readings can be used to illustrate the change of static pressure as velocity of flow changes.

Equipment. Glass apparatus is used as shown in Fig. 17. Potassium permanganate solution can be used, but ink or another colloidal suspension is preferable. Adjustment of air pressure over the colored liquid, or the height of the colored liquid reservoir, is important to insure good flow of colored liquid when water velocity is changed. The piezometer tube fluid may be mercury, but if the glass orifice is not small enough to give reasonable pressure differences in the tubes, a fluid lighter than mercury should be used. Carbon tetrachloride colored with a bit of dye makes the best manometer fluid when the pressure changes are small.

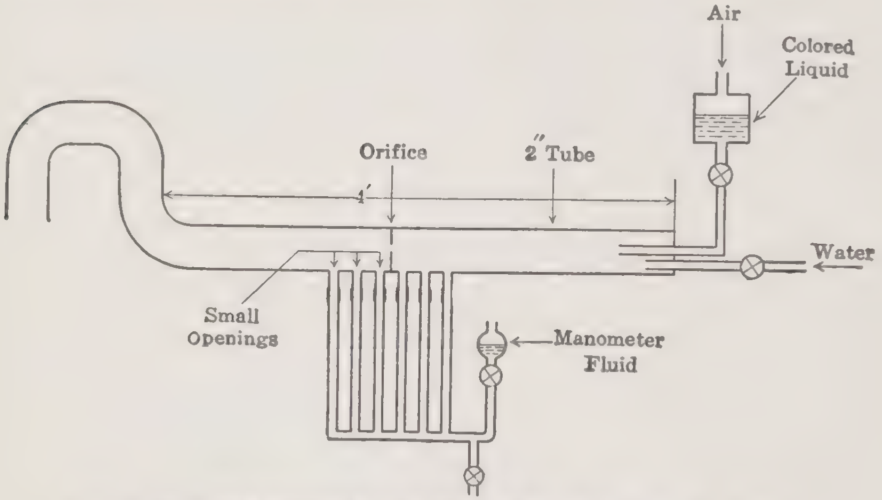


FIG. 17.—Glass apparatus for flow of water through an orifice.

Procedure. The piezometer tubes are half filled with liquid (mercury, carbon tetrachloride, or any suitable liquid). Water is then allowed to fill and flow through the system. Adjust the flow of colored solution so that the lines of flow can be observed. For various rates of water flow observe the behavior on each side of the orifice. Observe piezometer liquid heights.

To calibrate the orifice record the maximum pressure drop across the orifice and find the time required to discharge a definite weight of water. Repeat for at least six different rates of flow.

REPORT

1. Sketch lines of flow showing eddy currents on both sides of the orifice.
2. Plot pressure heads versus distance from orifice on each side of orifice, and draw the pressure gradient for each rate of flow.
3. Plot flow of water through the tube versus the maximum ΔH across the orifice.
4. Determine an equation and corresponding constant for the orifice from the data taken. Compare with the orifice equation and constant for the size orifice used.

EXPERIMENT NO. 10

FLOW OF WATER THROUGH FITTINGS

Object. 1. To determine the loss in fluid head for water flowing through pipe, fittings, and measuring devices.

2. To check observed values of head loss with values calculated from theory.

3. To determine total head loss due to a Venturi meter.

4. To determine equivalent frictional lengths of standard fittings.

5. To calibrate a Venturi meter and evaluate constants for its use.

6. To illustrate hydraulic gradient.

THEORY

Open piezometer tubes or manometers can be used to find static pressures at points through a pipe system provided the openings in the pipe are not rough and are sufficiently far from disturbances to eliminate false readings. The static head loss per foot of pipe due to friction for water flowing through each size of piping can be determined by liquid heights in the piezometer tubes attached to the piping. The head loss due to any fitting can be found experimentally by subtracting the head loss due to the length of pipe on each side of the fitting from the total head loss measured by the piezometer tubes on opposite sides of the fitting. These experimental values may be compared with accepted values for similar clean pipe fittings. Data may be obtained in terms of head of fluid flowing to calibrate the Venturi meter and also to determine the permanent head loss due to the meter.

Equipment. Apparatus as shown in Fig. 18 is used. The static heads at the indicated stations can be read by manometers or by open glass piezometer tubes. A plug cock valve should be used at the base of all piezometer tubes so that any one may be shut off from the system. It is important that the overflow on the constant head tank is sized large enough to take care of fluctuations in the tank. By inserting unions, or flange joints, various size piping can be substituted for those shown in the flow sheet.

Procedure. Fill the apparatus slowly with valve *B* closed. Be sure that all air has been displaced from the system and bring the level in the constant head tank to the desired height. Check each piezometer tube and be sure that all levels are at the same height as the level in the tank. If air is trapped in the system it can be detected easily by nonuniformity of static pressure. By running a small amount of water through the system trapped air can be eliminated through the tubes.

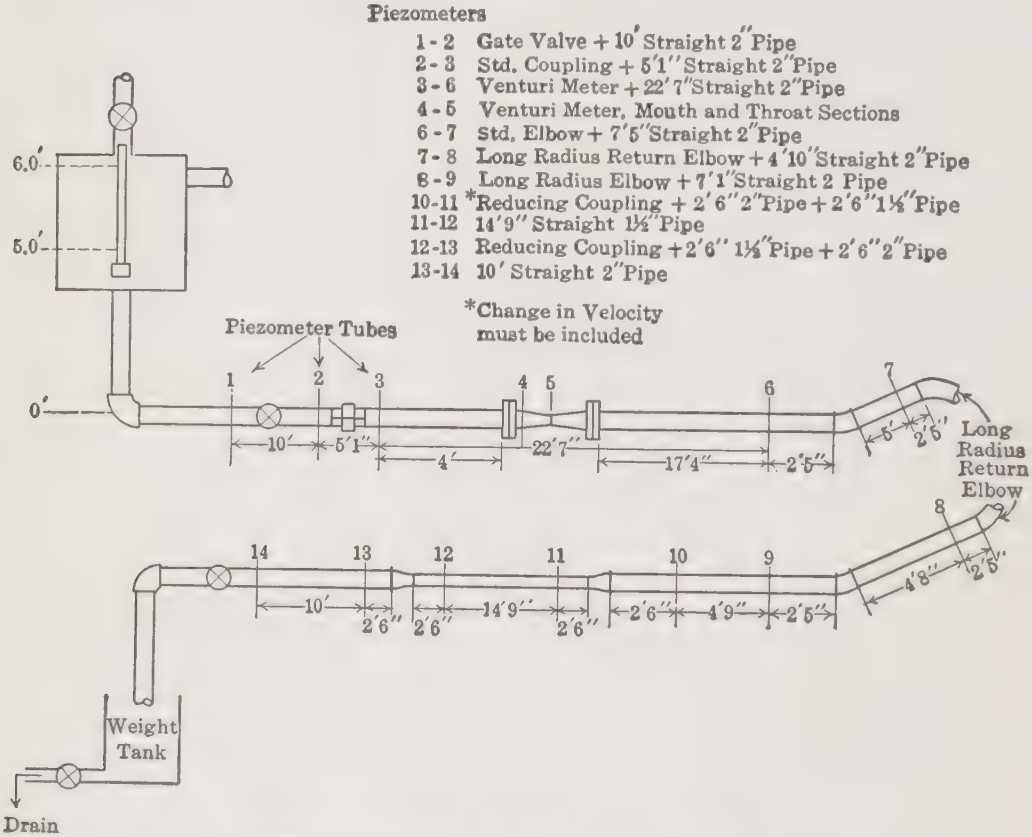

Piezometers
1-2 Gate Valve + 10' Straight 2" Pipe
2-3 Std. Coupling + 5'1" Straight 2" Pipe
3-6 Venturi Meter + 22'7" Straight 2" Pipe
4-5 Venturi Meter, Mouth and Throat Sections
6-7 Std. Elbow + 7'5" Straight 2" Pipe
7-8 Long Radius Return Elbow + 4'10" Straight 2" Pipe
8-9 Long Radius Elbow + 7'1" Straight 2 Pipe
10-11 *Reducing Coupling + 2'6" 2" Pipe + 2'6" 1½" Pipe
11-12 14'9" Straight 1½" Pipe
12-13 Reducing Coupling + 2'6" 1½" Pipe + 2'6" 2" Pipe
13-14 10' Straight 2" Pipe
*Change in Velocity must be included
6.0'
5.0'
0'
Piezometer Tubes
1
2
3
4
5
6
7
8
9
10
11
12
13
14
10'
5'1"
4'
22'7"
17'4"
2'5"
5'
2'5"
Long Radius Return Elbow
4'8"
2'5"
10'
2'6"
2'6"
14'9"
2'6"
2'6"
4'9"
2'5"
Weight Tank
Drain

Fig. 18.—Equipment to study flow of water through fittings.

To operate, control the flow by valve *B*, always holding constant level in the overflow tank. Be careful never to run at such a rate that any air is sucked in through a piezometer tube. The limit of flow through the Venturi meter is reached when the throat tube level is at the bottom of the glass tube. For higher flows through the meter, either close the cock at the throat connection or use a manometer across the Venturi connections.

Make five runs at different rates of flow, starting with a rate at the limit of the Venturi throat tube. Decrease the velocity for successive runs. Weigh at least 300 pounds of water for each run, find the time required, and repeat the weight determination as a check run. During each run record the height of water in all piezometer tubes. Take the temperature of the water.

Make three more runs, increasing the rate of flow up to the limit of the last tube. In such runs the Venturi throat connection must be closed or a mercury manometer of sufficient size used across the meter.

REPORT

1. Plot an hydraulic gradient for the system for one rate of flow.
2. Calculate the head loss per foot of pipe for each rate of flow for both sizes of pipe used. Plot these values against velocity of flow.
3. Calculate the head loss due to each fitting for each rate of flow and plot the values against velocity.
4. Calculate the head loss due to each fitting in terms of equivalent lengths of straight pipe and compare with values in handbooks.
5. Calculate the Reynolds number for each flow in both sizes of pipe. From these and the Fanning plot, Fig. 19, calculate the friction head loss per foot of pipe and compare with observed values.
6. Draw a calibration curve for the Venturi meter.
7. From log-log plots evaluate the equation for the Venturi meter when the discharge is measured in feet per second, in gallons per minute, and in cubic feet per minute.
8. In the equation G.P.M. $= K\sqrt{\Delta H}$, where $K = C_v K'$, calculate the value of K for a Venturi meter whose dimensions are: $I.D. = 2.067$ inches, throat diameter $= 1.25$ inches, and $C_v = 0.98$.

EXPERIMENT NO. 11

Flow of Fluids Through a Heat Exchanger

Object. 1. To determine head loss for fluids flowing through a heat exchanger.

2. To determine power requirements for pumping fluids through equipment utilizing odd shapes of fittings and arrangements.

THEORY

The overall pressure drop of water flowing through the annular spaces of a double pipe heat exchanger connected by means of ammonia flanges or other special fittings can be determined by static pressure connection

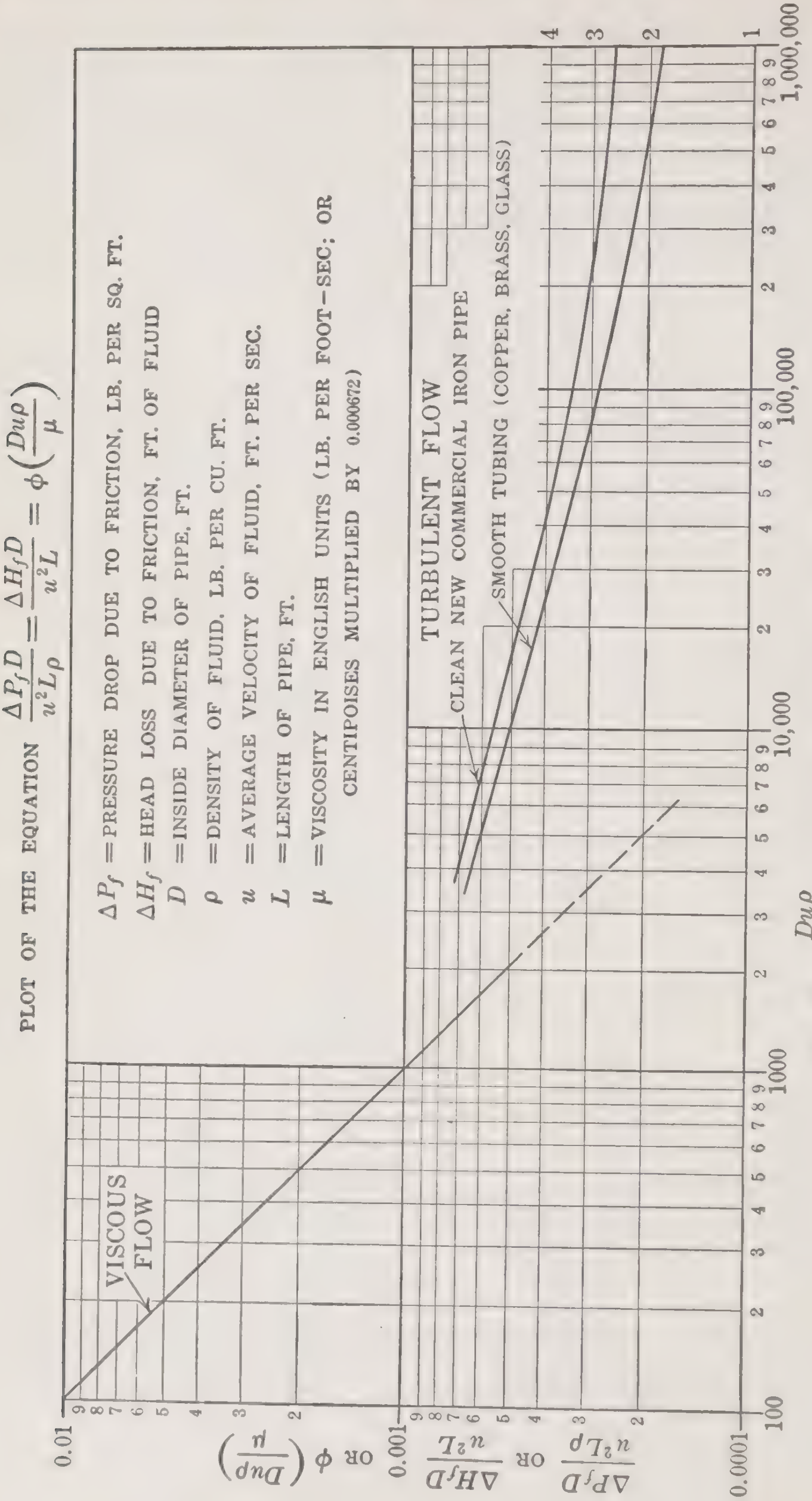


FIG. 19. — $\frac{Du\rho}{\mu}$ FRICTION OF FLUIDS IN PIPES

to manometers. By measuring the pressure drop across one straight section of the annular space the drop per foot of length can be determined. From such data the head loss due to the resistance of the fittings used can be found.

By the use of Reynolds number the data obtained for water may be used for other fluids at various temperatures. When the Fanning relation does not hold due to unclean pipe, the Reynolds number may be modified by a constant factor so that ΔH_f, from Fig. 19, can be checked and variation due to roughness accounted for.

Equipment. A diagram of the apparatus is shown in Fig. 20. The flow through the annular space can be measured by the orifice shown.

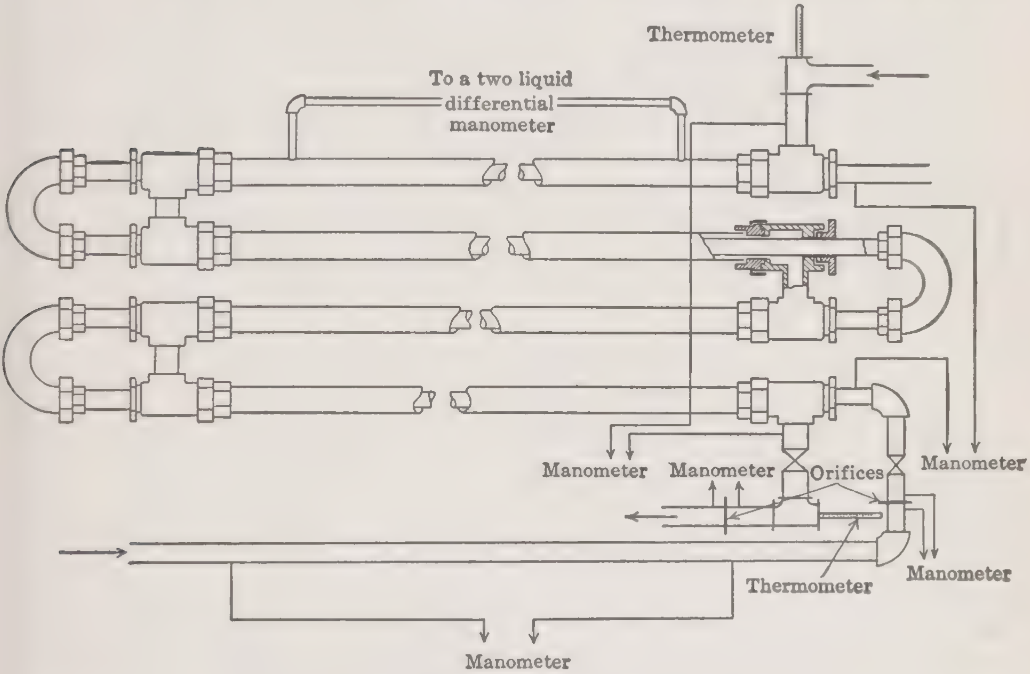


Fig. 20.—Flow of fluid through heat exchanger.

By simple rearrangement the inside piping and connections may be used in the experiment as well as the annular space. Carbon tetrachloride is used in manometer B, which gives the head loss in a small straight section of the annular space. Control of the flow should be made by valve C.

Procedure. Fill the apparatus with water and see that all manometer tubes and lines are filled with liquid. Take readings of pressure drop across the entire exchanger and across a straight section. Take data for the rate of flow and measure the temperature of the water. Make five runs at different rates of flow. As the experiment progresses make a laboratory plot.

REPORT

1. Find the head loss due to friction for each flow for one foot of pipe.
2. Determine the equivalent length of pipe for a single ammonia fitting (or other type of fitting used).
3. Compare the value ΔH_f for the straight annular space calculated from the *Re* and Fig. 2 with the experimentally determined value.
4. A double pipe heat exchanger is to be installed with piping and fittings of the same size and roughness as the present equipment, but having four more passes and with each pass 5 feet longer. The exchanger is to be used to cool aniline from 150° F. to 70° F. and which will flow in the annular space. Determine the yearly cost for pumping the aniline through the heat exchanger. Assume 350 operating days of 24 hours each, per year; power at 1.8¢ per K.W.H.; overall pump and motor efficiency of 55%.

It may be necessary to use the exchanger for cooling castor oil under the same conditions. Over what range of operation will the data for aniline apply to castor oil?

EXPERIMENT NO. 12

PUMP TEST AND CALIBRATION OF METERS

Object. 1. To determine the performance of a pump for various discharge pressures and rates.

2. To illustrate the operation of measuring instruments and their auxiliary equipment.

3. To calibrate fluid flow meters and to derive equations for their use.

4. To compare the sensitivity and range of usefulness of different types of measuring devices.

5. To compare the head loss of the different meters.

THEORY

To determine the efficiency of a pumping set-up and to find the performance of a pump under different conditions, it is necessary to measure the power input to the pump and to determine by means of a flow meter and a pressure device the work done by the pump.

To calibrate and compare metering instruments fluid may be allowed to flow through the various instruments in series and finally into a weight tank where an absolute determination of the rate of flow can be made. To calibrate meters and to develop equations for their use it is necessary to plot data and by means of these plots to find empirical equations. These may be compared with the standard equations for use with such meters.

Equipment. Fig. 21 is a diagrammatic flow sheet of the equipment used for the experiment. When a pump test is to be run, the valves are so manipulated that the constant head tank is removed from the system and the discharge head regulated by the control valve. A pump test may also be run with discharge into the constant head tank. The apparatus may be arranged so that different levels in the constant head tank may be maintained. A voltmeter and ammeter should be available to measure the power used to operate the pump motor. If the pump used is a steam pump, a condenser is provided so that the steam can be condensed and weighed to determine the quantity of steam used.

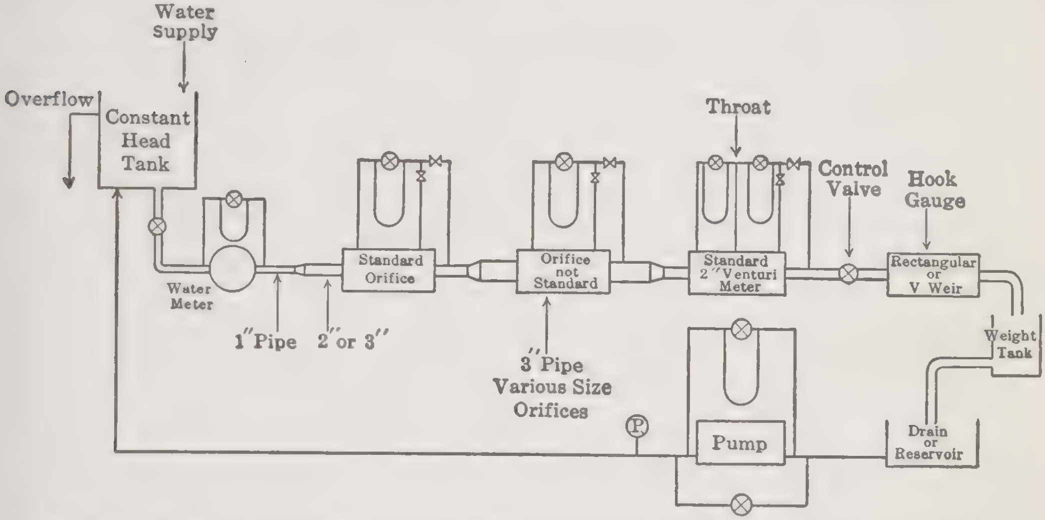


FIG. 21.—Diagrammatic flow sheet of the equipment to be used in Experiment 12.

The flow sheet shows static pressure connections to the U-tube manometers in addition to those required for measurement of flow and are for the purpose of measuring the permanent head loss due to the resistance of the instrument. During operation either the constant head tank may be used or flow produced by the pump without the use of the constant head tank.

Procedure. *Pump Test:* Using a centrifugal or rotary pump, having a D.C. electric motor drive, connect an ammeter and voltmeter to measure power input. Pump water at a known temperature through any part of the system desired, and control the pressure on the discharge of the pump by means of the control valve or the level in the constant head tank. Determine the head at the discharge of the pump and the head loss through the entire discharge side of the system. Measure the flow of water either by direct weight or by flow through one of the calibrated instruments. Repeat for at least four different heads and different rates of flow.

If a reciprocating steam driven pump is used, condense the exhaust steam and weigh it to determine the amount of steam used in a definite period of time. Control the rate of flow by the piston speed or by the length of stroke. Record the steam pressure and the pressure at the discharge of the pump. Also determine the total pressure drop through the system through which the water is pumped. Otherwise perform the experiment as outlined above.

Calibration of Meters. The various meters can be calibrated during the pump test, or separately if desired. They should be calibrated over the whole range of the instrument. Care should be taken so that if a higher flow is desired than can be handled by any one of the instruments, the manometer connections to that instrument are closed. Make at least five runs covering the range of the manometer on each instrument. Read manometer heights. Weigh at least 200 pounds of water for each determination of rate of flow. Record the water temperature. For each run also read the manometers when connected to give permanent head loss across the meter. During the performance of the experiment make a laboratory plot as described in Experiment 7.

REPORT

1. Determine the overall pump efficiency at different rates of flow and at different discharge pressures. Plot efficiency versus discharge pressure for each rate of flow.
2. Calculate the velocities in feet per second and gallons per minute from the weight determination. Plot a calibration curve for each instrument in terms of velocities versus heads, and also in terms of gallons per minute versus heads.
3. From appropriate plots evaluate an equation for use with the orifice, the Venturi meter, and the weir in the set-up, and compare these with their corresponding equations in the texts.
4. Calculate the permanent head loss in terms of per cent of upstream static head for each instrument and compare them.
5. Tabulate and compare the range of usefulness, behavior, adaptability, and other characteristics of each measuring device.
6. From results of these experiments and tables in handbooks, estimate the cost for pumping 5 gallons per minute of a 20% sugar solution through 200 feet of piping (of the same roughness as that used in the test) to an elevated tank 20 feet above the discharge of the pump, where the level in the tank is maintained by means of an overflow at

10 feet from the bottom of the tank. Include in the 200 feet of pipe line one of the orifices which has been calibrated. Assume the cost of electricity to be 2.0¢ per K.W.H. if electricity is used, and for steam 1.0¢ per thousand pounds at 125 lb. per sq. in. gauge.

EXPERIMENT NO. 13

Flow of Air

Object. 1. To illustrate the character of air flow in a round or rectangular duct by means of a Pitot tube.

2. To make a pipe traverse.

3. To operate a Pitot tube and a draft gauge.

4. To determine the head loss due to friction for air flowing through a duct.

5. To determine overall blower efficiency.

THEORY

Direct measurement of the velocity of flow of air can be made by means of a Pitot tube connected to a draft gauge. Velocities calculated from Pitot tube readings can be plotted against the distance along any diameter of the duct to give a graphical representation of the character of the air flow. By means of such a plot it is possible to determine whether the flow of air is in viscous or turbulent motion. If the major portion of the cross-section has nearly the same velocity of flow, then the flow is turbulent. If the velocity of the flow increases rapidly all the way to the center of the duct and then decreases to the opposite side, the flow may be said to be viscous. By making velocity determinations at the proper traverse points, i.e., at the center points of equal area, it is necessary only to average the traverse readings arithmetically to obtain the average velocity across one diameter.

By measuring the static head loss over a long section of the duct the friction head can be determined.

The power output of the blower can be determined by measuring the flow and pressure of the air, knowing its density. Power input to the motor driving the fan can be determined by voltage and amperage measurements.

Equipment. Apparatus is shown in Fig. 22. The blower may be connected directly or by means of a pulley to a D.C. motor. The rate of flow of air can be regulated either by changing the speed of a D.C. motor or by using an adjustable pulley set so that a constant speed motor may be used for variable speed blower operation. At least twenty feet of straight duct should be used. This duct may be either round or rectangular. The screen near the blower is for the purpose of smoothing out the flow of air and for breaking up swirls of air. The screen at the end of the duct is used to build up a static pressure at that end. By using several different mesh screens the static pressure may be varied considerably. Ordinary wire screens having openings ⅜ inch square will produce considerable static pressure. A draft gauge or a two liquid differential manometer can be used with the Pitot tube and also at points marked for static pressure readings. The Pitot tube should be detachable so that it can be used across several different diameters of the duct.

Procedure. Determine the points along a diameter at which readings should be taken for a ten point traverse. If a rectangular duct is being used, determine the points at the center of equal area rectangles where

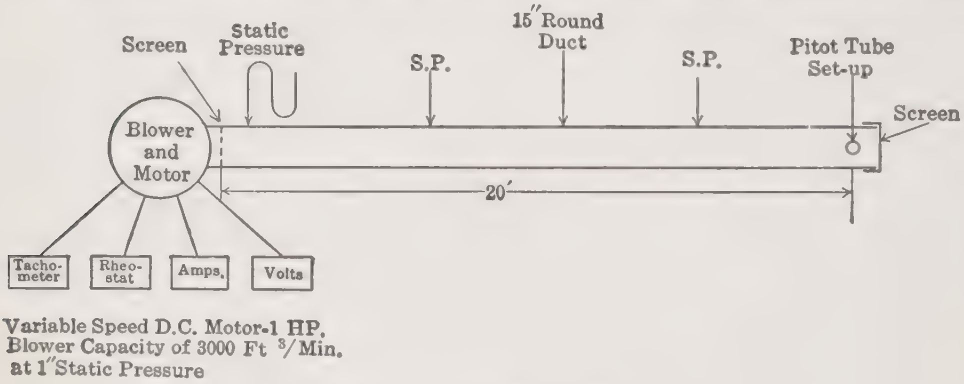


FIG. 22.—Flow of air, Experiment No. 13.

Pitot tube readings are to be taken. Connect an ammeter and voltmeter in the motor circuit. Fasten a screen at the end of the duct and place a Pitot tube in the center of the duct. Connect a draft gauge to the Pitot tube as shown in Fig. 8. Run the blower at such a speed that the draft gauge reading for impact head is at the limit of the draft gauge. Measure the speed of the blower by means of a tachometer. Maintain the same speed throughout any one run. Move the Pitot tube along one diameter and take readings at each point of a ten point traverse and also at the center of the pipe. Make both impact and static pressure readings for each position of the Pitot tube.

Connect a draft gauge or two-liquid differential manometer to the static pressure opening nearest the blower, and make a reading for the pressure at this point. Repeat for any other static pressure openings desired. Record tachometer readings and ammeter and voltmeter readings.

Hold a sling psychrometer at the end of the duct to determine the wet and dry bulb temperature of the air. Determine the density of the air from a humidity chart.

Repeat these traverses along at least one other pipe diameter.

Repeat these observations for three other lower blower speeds.

REPORT

1. Plot the static pressure distribution across the duct for each run.
2. Plot the velocity distribution across each diameter traversed for each run.
3. Determine the average static pressure for each rate of flow, and the average velocity for each rate of flow.
4. Plot center velocity against average velocity for each flow.
5. Calculate the H.P. developed by the blower for each rate of flow, and plot against blower speed.
6. Calculate the H.P. input to the blower system and overall efficiency. Plot efficiency against blower speed and air H.P. developed.
7. Determine the friction head loss per foot of duct for each rate of flow.

EXPERIMENT NO. 14

Measurement of Gas Flow

Object. 1. To operate different gas measuring instruments and the necessary auxiliary equipment for them.

2. To calibrate gas measuring instruments.

3. To compare the different instruments and methods as to accuracy and usefulness.

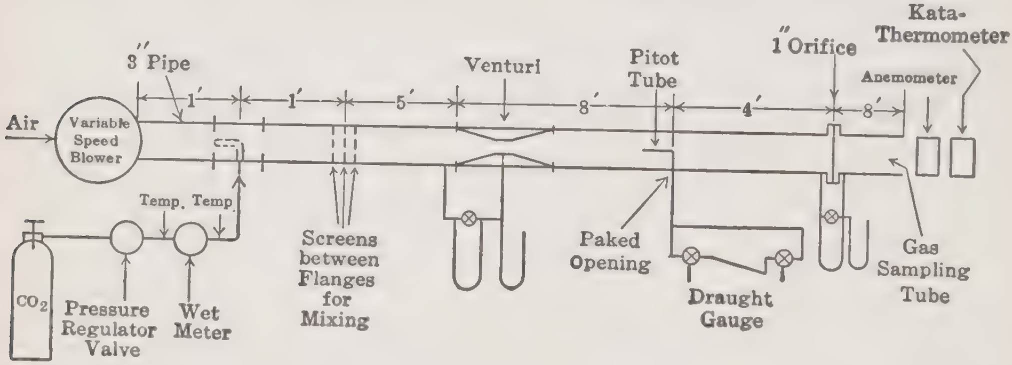
THEORY

The rate of flow of gas through a duct can be most accurately measured by means of a Pitot tube. Using Pitot tube readings as a base, other measuring instruments can be calibrated with reference to it. It is important that the location of the Pitot tube be such that there are no disturbances of flow near it. Complete traverses of the duct should be made with the Pitot tube before using it as a basis of comparison for the other instruments.

For dilution metering a wet gas meter can be used to measure a constant flow of carbon dioxide injected into the gas stream where it is mixed thoroughly.

Equipment. Diagram of the apparatus is shown in Fig. 23. A long straight section of pipe is fitted up with the various instruments as shown. It is important that thorough mixing of CO_2 with the flowing gas is obtained in the case of metering by dilution. Analyze for CO_2 by an Orsat apparatus, or an automatic recording CO_2 meter. The vane anemometer should be the proper size for the pipe. The Kata-thermometer or a hot wire anemometer can be located in any convenient place. The blower is connected to a variable speed motor and a volt meter and ammeter connected so that power consumption can be measured.

Procedure. Operate the blower for runs at five or more different flows of air, and make a determination of the air flow with each instrument for each run. For the dilution method, supply CO_2 at a constant rate through the wet meter so that its rate of flow is at least 10% of the total gas flow in the test pipe. Allow CO_2 to run into the pipe for at least 15 seconds before sampling the gas to analyze for CO_2. Analyze a sample of inlet air (at the blower) and make two analyses of the mixed gases at the end of the system for each run. Appropriate corrections for volume of CO_2 measured by the wet meter must be made, based upon the pressure of gas in the wet test meter and the saturation of the gas by the water in the meter at its temperature.



Blower-125 Ft. ^{3}/Min. at $\frac{3}{8}''$ S.P.
American Blower Co. - Baby # 2

FIG. 23.—Apparatus for measurement of gas flow.

Inclined manometers, draft gauges, or two-liquid differential manometers can be used with the Venturi meter, the orifice, or the Pitot tube. Static pressure readings can be made by proper valve manipulations on the lines connecting the manometers to static pressure connections.

The vane anemometer should be held directly against the end of the pipe or a fixed distance from the end of the pipe and a stop watch used for timing. The Kata-thermometer or a hot wire anemometer can be held just outside the pipe.

The temperature of gas flowing should be measured for all runs. Wet and dry bulb readings are also to be taken in order to determine the density of inlet air.

If the limit of any measuring device is not reached in the five runs, disregard the other instruments and make a series of determinations with flows approaching the limit of the particular instrument in question.

REPORT

1. Determine the rate of flow in cu. ft. per minute for each run by the Pitot tube and by each of the other methods used.
2. Tabulate a comparison of the cu. ft. per minute of air flow for each run by each method.
3. Plot cu. ft. per minute by the Pitot tube versus cu. ft. per minute by each of the other methods. .
4. By a log-log plot find an equation for use with the orifice used, and compare the constant with accepted orifice coefficients.
5. Discuss apparent inaccuracies and limits of usefulness of each method used.

EXPERIMENT NO. 15

Measurement of Steam Flow

Object. 1. To measure flow of steam by means of an orifice.
2. To compare different types and sizes of orifices.
3. To compare results with theoretical formulas.

THEORY

Steam flow may be measured by an orifice similarly to the measurement of other fluid flows. To calibrate an orifice for steam a condenser is used to condense the steam after passing through the orifice. The condensate is weighed, and the rate of steam flow can be calculated. Since steam is compressible, the simple equation for flow through an orifice will hold only over small ranges of pressure drop. Formulas are referred to in the introduction of this chapter which account for compression and expansion of fluids while passing through an orifice meter.

Equipment. Diagram of the apparatus is shown in Fig. 24. Pressures may be measured either by means of pressure gauges or by mercury manometers. The condenser must be sufficiently large to insure complete condensation of the steam. At least two different sized orifices are to be used. In addition, both a sharp edged and a round edged orifice are to be used. Control is made principally through valve *B*.

Procedure. Place a sharp edged orifice in position. Allow steam to flow through the equipment slowly until it is completely warmed.

Place calibrated 150 lb. pressure gauges in place as shown. Operate values *A* and *B* so that upstream pressure is maintained at 125 lbs. gauge. Open valve *B* until the maximum pressure difference is observed on the two gauges. When conditions are steady find the time required for at least fifteen pounds of steam to pass through the orifice. Repeat at 5 uniform decrements of pressure drop and draw a laboratory plot of pounds of condensate per minute versus pressure drop. Repeat any runs which seem necessary.

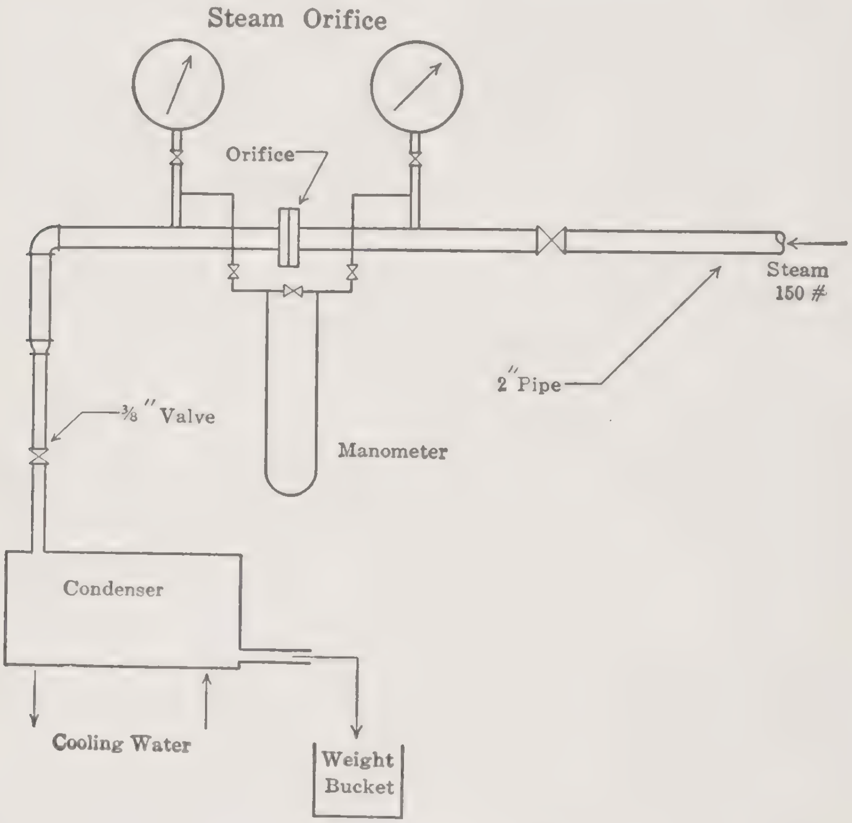


FIG. 24.—Measurement of steam flow.

Place another sized orifice in the line and repeat the experiment.

Place a round edged orifice in the line and make a series of experiments with the round edge upstream and another series of experiments with the round edge downstream.

For low rates of steam flow, place a small orifice in the line and use a mercury manometer to indicate pressure drop across the orifice. Run a set of experiments as described above. If it is desired to measure a large steam flow by means of the manometer, use a larger orifice in the line.

When using the manometer, use gauge C to indicate line pressure. This pressure should be maintained constant in any one set of runs.

REPORT

1. For each orifice used plot pressure drop versus flow of steam.
2. For each orifice plot pressure drop versus flow on log-log or semi-log paper and determine an empirical equation to be used for the particular orifice. Indicate the limits of usefulness of these equations.
3. For each orifice used describe its characteristics and state the limits of its usefulness.
4. Should a round edged orifice be used with the round edge upstream or downstream?
5. For each orifice calculate the flow from recommended formulas within the limits accepted for these formulas and compare the results with the actual test data.

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Chapter III

FLOW OF HEAT

H. McCormack[1]

A mathematical study of the flow of heat was not possible until two assumptions had been made and generally accepted.

The first assumption is that the transfer surface intervening between two spaces, from one of which heat is being transferred to the other, is covered on each side by a film having different properties from the main streams of material between which the transfer is occurring.

This assumption arose from experimental study of heat transfer through metal walls, the experimental data showing rather definitely resistances to transfer far in excess of that which could be predicted from the conductivity of the intervening wall.

These layers of material are referred to as films and the theory named the film concept of heat transfer.

The second assumption bearing on the calculations for flow of heat, arose from this film concept of transfer. It was assumed that, with these films present on the transfer surface, the flow of heat through the films must be analogous to other types of fluid flow and on this basis of similarity, formulas were developed for flow of heat on a basis comparable to the formulas developed for the flow of fluids.

These assumptions have particular relation to flow of heat by convection and have no relation to flow of heat where the flow is solely by conduction, nor to conditions where the flow of heat is solely by radiation. There are, however, extremely few instances in which the flow is solely by conduction or solely by radiation.

There are no instances, in mind, where the flow does not occur through a retaining wall, and of course, each surface of the retaining wall has a film through which the transfer must take place. This, however, does not apply to a solid heated entirely by radiation.

A brief statement as to the conditions under which these various types of heat flow occur seems to be in order.

Heat transfer by conduction takes place when there is a difference in temperature between points on the same body or when two different bodies at different temperatures are in contact.

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It is inconceivable, however, that two bodies may be in such intimate contact, that no film intervenes between the two adjacent surfaces. Heat transfer by conduction is dependent only upon difference in temperature, the dimensions of the bodies and the physical properties of the bodies. Heat transfer by conduction involves, therefore, only the questions of temperature difference, thickness of the bodies through which the transfer is occurring, and the thermal conductivity of this body.

Heat transfer by convection occurs in gases and liquids. It is essentially a fluid flow where new particles of the gas or liquid are constantly coming into contact with the heat transfer surface and imparting or receiving heat from this surface. It is obvious that any condition tending to increase the gas or liquid velocity will increase the rate of heat transfer. Two transfers by convection are recognized—forced convection where the movement is induced by mechanical methods, and free convection in which the movement is due to temperature differences.

Radiation occurs when two surfaces at different temperature levels are "visible" to each other and separated only by a medium through which radiation can pass. Transfer by radiation is, therefore, limited to gas filled spaces. The transfer through a gas filled space is dependent, to a considerable extent, on the properties of the gas through which the radiation is passing. There is considerable difference in the "carrying" capacity of the different gases.

Each of these three types of heat flow will be treated experimentally with major emphasis, however, on transfer by convection. This, on the basis that the majority of the industrial operations in which flow of heat is important, is conducted in a manner to bring about transfer by convection. A minor consideration in this emphasis of heat flow by convection is that the mathematics of heat flow by convection has received much more detailed study, and has required much more detailed study, than transfer by either of the other methods.

The following exposition of the development of the general equation for flow of heat by convection is quoted from "Industrial Heat Transfer," by Schack.[1]

"The most essential application of the theory of similarity to heat transfer is that the temperature fields of two streams will be similar when VD/a has the same value for each stream. This will be referred to again subsequently. For the present it is sufficient to observe that when the values of VD/a are equal, the values of hD/k must likewise be equal. In other words, when two streams are similar, the expressions hD/k and VD/a will each remain constant in spite of individual variations in a, V, D, h, or k. Moreover, any other stream, not similar to those just considered, will have a different value for VD/a which will necessitate a different value for hD/k. It can be shown that for each value of VD/a there is a definite corresponding value of hD/k, which, in mathematical terms, is expressed as;

$$\frac{hD}{k} = f\left(\frac{VD}{a}\right) \tag{151}$$[1]

The form of the function, f, must be determined by experiment. Equation 151 greatly facilitates investigation, however, since it is only necessary to change one variable to determine f.

The manner in which f is obtained may best be illustrated by reference to the historical development of the theory. Nusselt developed Equation 151 and found that, for a brass pipe with an inside diameter of 0.87 in., the surface conductance h increased with the 0.786 power of the velocity, V, when the other variables remained unchanged, or

$$h = C_1V^{0.786} \tag{152}$$

where C_1 represents all variables unchanged during the test. From Equation 151 it follows that;

$$h = \frac{k}{D}f\left(\frac{VD}{a}\right) \tag{153}$$

Setting Equations 152 and 153 equal to each other with V as the only variable yields

$$\frac{k}{D}f\left(\frac{VD}{a}\right) = C_1V^{0.786} \tag{154}$$

This equation is satisfied for all values of V only if

$$f\left(\frac{VD}{a}\right) = C\left(\frac{VD}{a}\right)^{0.786} \tag{155}$$

since V must appear in the same form on each side of the equation, that is, as $V^{0.786}$, in order to insure equality. Owing to the relation of D and a to V, expressed by the quantity VD/a, the form in which D and a appear is likewise determined, and the form in which k appears is established by Equation 153. The advantage of the theory of similarity is evident in this simultaneous determination of the form in which D, a, and k appear from the single experimental determination of the influence of change in V.

Substitution between Equations 155 and 153 yields

$$h = \frac{k}{D}C\left(\frac{VD}{a}\right)^{0.786} \tag{156''}$$

Various modifications of and deductions from this general formula have been developed and applied to particular conditions under which flow of heat by convection is occurring. These formulas will be given and commented on when studying the particular conditions under which they are applicable.

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[1] The formula numbers are as in the quoted reference.

Heat transfer by radiation—The fundamental formula for flow of heat by radiation has been referred to in the consideration of radiation pyrometers. It is the equation based upon the Stefan-Boltzmann law and can be expressed as follows:

$$q = CA\left[\left(\frac{T_1}{100}\right)^4 - \left(\frac{T_2}{100}\right)^4\right] \text{ B.t.u. per hr.} \qquad (305)$$

In this equation

C = radiation constant, B.t.u. per ft.^{2} per hr.;
A = area of radiating or irradiated surface, ft.^{2};
T_1 = temperature of the radiating surface, R;
T_2 = temperature of the irradiated surface, R.

The radiation constant C for many solids used industrially has been determined and can be found in tables of physical constants. A factor frequently giving trouble, in industrial calculations involving flow of heat by radiation is uncertainty in determining the area "visible" to the radiating body.

The radiation law of Lambert is of assistance in computing heat transfer by radiation under such conditions.

"The radiation from a surface in a direction at an angle with the surface varies as the cosine of the angle between the direction of radiation and the normal to the surface. If this angle is ϕ, the radiation in the given direction is

$$q = q_n \cdot \cos\phi \text{ B.t.u. per hr.} \qquad (307)^*$$

where q_n is the radiation normal to the surface." Lambert's radiation law is only an approximation for many bodies, and then only when the surfaces radiate diffusely. A diffused radiation is sent out by a body which does not reflect incident light as a mirror does, but disperses the light in all directions as, for example, a dull white chalk surface. The diffused dispersion of the light is caused by the minute irregularities of the surface, which form small mirrors placed irregularly facing in all directions. Lambert's law does not hold for polished metals, which, on the contrary, generally show greater radiation in an oblique direction than perpendicular to the surface.

Integration of Lambert's equation gives

$$q_n = q_{total} \text{ B.t.u. per hr.} \qquad (308)^*$$

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* Numbers of formulae are from reference (1).

for the radiation normal to the surface. q_{total} is the total radiation as given by Equation 305, the Stefan-Boltzmann law. Equation 308 therefore becomes

$$q_n = CA \frac{\left[\left(\frac{T_1}{100}\right)^4 - \left(\frac{T_2}{100}\right)^4\right]}{\pi} \text{ B.t.u. per hr.} \tag{309}$$

The value of q_n from this equation is substituted in Equation 307.

The intensity of radiation from a point source of radiant energy decreases with the square of the distance from the point source, or, in mathematical terms,

$$I = \frac{I_1}{r^2} \tag{310}$$

where I_1 is the intensity at one-foot distance and r is the distance in feet."

Many of the operations, in chemical engineering, in which the flow of heat is an important factor, consist of somewhat complex transfers where the flow through two or more materials is involved.

Thus in a heat exchanger there is involved the transfer through the fluid film on the hot side, the transfer through the intervening wall, and the transfer through the fluid film on the cold side.

Here the theory of similarity is again of assistance, the relationship now being that between the flow of heat and the flow of electricity. As the total resistance in an electric circuit is the sum of the individual resistances, so also is it in a "heat circuit," and the conductivity in each of the cited instances is the reciprocal of the resistance or resistances, as the case may be.

The general formula for heat flow by conductance may be expressed as:

$$Q = kA \frac{dt}{dL}$$

where k = thermal conductivity in B.t.u. per sq. ft. per foot of thickness per hour;

A = area in sq. ft;

dt = temperature change in passing through the infinitesimal distance dL; therefore

$\frac{dt}{dL}$ = temperature gradient.

Derived from this equation, by integration, is one giving the conductance through a plane wall:

$$Q = kA \frac{t_1 - t_2}{L} \text{ per sq. ft. per hr.}$$

and one giving the conductance through the exterior of a pipe surface:

$$Q = \frac{0.869k}{De \log \frac{De}{D_1}} (t_1 - t_e) \text{ per sq. ft. per hr.}$$

The formula for conductance through composite walls is given as illustrative of the method employed in treating series resistances; it is:

$$Q = \frac{(At_1 - t_n)}{\frac{L_1}{k_1} \frac{L_2}{k_2} \frac{L_3}{k_3} \cdots \frac{L_n}{k_n}} \text{ B.t.u per sq. ft. per hr.}$$

t_1 = temperature of the outside of the first wall;
t_n = temperature of the outside of the last wall;
A = area in square feet through which heat is flowing.

Here the resistances have been a series of *walls*; the method for solving would be the same were the resistances a combination of wall resistances and *film* resistances; substituting the reciprocal of the film coefficient as a *resistance* in the formula just stated.

A series of experiments have been selected, which, so far as possible, are illustrative of varying types of flow of heat and operated in such a manner as to permit the calculation of film coefficients at the heat transfer surface, together with convection, conduction and radiation factors.

The fundamental bases for the required calculations are the formulas which have been developed from the film concept for flow of heat by convection, the formulas for flow by radiation and the experimental values which have been determined as the radiation constants and the conductivity for various construction materials. These later values are to be found in the tables of physical and chemical constants.

Consider the simplest condition under which the film concept for the flow of heat applies, such as two liquids on opposite sides of a retaining wall with the liquids at different temperatures. Heat flows from the liquid having the highest temperature to and through the film on this side of the retaining wall, through the retaining wall, to and through the film on the opposite side of the retaining wall and into the liquid at the lower temperature. The films on the retaining wall vary in thickness depending on:

The smoothness of the surface of the retaining wall
The velocities of the two liquids past the surface of the retaining wall
The viscosity of the liquids

The quantity of heat flowing is also influenced by the area through which the liquids flow and the position of the retaining wall, whether horizontal, vertical, or at an angle with the horizontal.

The formulas developed to express the value of the film coefficients attempt to take account of all these variables.

The experimental development of these formulas for film coefficients has been commented on from the viewpoint of similarity and the theoretical development of the formulas occurring in this manner.

The formulas, when developed, were checked experimentally, utilizing the fact that the quantity of heat flowing from position A to position B is determined by the temperature difference and the amount of resistance interposed between A and B. Therefore, with a given temperature differential, the quantity of heat flowing is inversely proportional to the interposed resistance, and these resistances vary directly as the temperature drops at the points of heat transfer. Suppose, for example, in the case which has been cited, the temperature of liquid A is 60° F., the temperature of the adjacent side of the retaining wall is 66° F., the temperature at the distant side of the retaining wall is 67° F., and the temperature of liquid B is 90° F.

The quantity of heat flowing per sq. ft. per hour per 1° F. is 200 B.t.u. The temperature differences are:

Liquid A to retaining wall......	6° F.,	t_1
Through the retaining wall.....	1° F.,	t_2
Liquid B to retaining wall......	23° F.,	t_3
Liquid A to liquid B...........	30° F.,	Δt

Let t = the temperature difference, 30° F.
R = the total resistance.
r_1 = resistance liquid one to adjacent retaining wall.
r_2 = resistance of the metal tube.
r_3 = resistance tube wall to liquid two.
k_1, k_2, k_3 = the corresponding conductivities.

Then

$$200 = \frac{30}{R}, \quad R = 0.15$$

$$r_1 = .15 \times \frac{6}{\cancel{15}\ 30} = .030$$

$$r_2 = .15 \times \frac{1}{30} = .005$$

$$r_3 = .15 \times \frac{23}{30} = .115$$

and

$$k_1 = \frac{.15}{.030} \times 200 = 1000$$

$$k_2 = \frac{.15}{.005} \times 200 = 6000$$

$$k_3 = \frac{.15}{.115} \times 200 = 260.8$$

It is thus seen that when the temperatures of the various surfaces at which transfer of heat occurs are known, it is possible to calculate the film resistances and the film conductivities *without reference to any formulas for film coefficients.*

This fact is utilized in checking the accuracy of any developed formula; the numerical value obtained by using the formula must check with the numerical value calculated from the temperature differences. The formulas are of most value when it is difficult or impossible to determine the temperatures at all surfaces of a system through which heat is flowing, and when designing equipment to operate under certain assumed conditions.

A series of experimental studies of flow of heat have been chosen to cover as completely as possible, the various ways in which heat may flow. Much of this study, of necessity, calls for particular laboratory equipment and could not be conducted in available industrial equipment.

A factor necessary to any accurate mathematical study of flow of heat is a reliable method for measuring temperatures at desired points in the system through which heat transfer is occurring.

Comments regarding thermocouple installation insulation, calibration and methods of operation in specific cases have been cited in a previous chapter. (See pages 61-62.)

Recent experimental studies indicate that assumptions as to temperature uniformity in certain systems must be abandoned and provisions made to take temperatures at distributed points in the system. Illustrating this: It has been found that the temperature varies, around the circumference of a horizontal pipe upon which vapors are condensing, or over which hot gases are flowing—particularly in a staggered tube bank—and from top to bottom of a vertical tube or wall.

EXPERIMENT NO. 16

Radiant and Convection Transfer to Air in Motion

REFERENCES

McAdams, Heat Transmission, pp. 50-86, 135-152, 169-179.

Walker, Lewis, and McAdams, Principals of Chemical Engineering, pp. 49, 63, 71, 149, and 166-169.

Badger and McCabe, Elements of Chemical Engineering, pp. 46-48, 116-159.

Haugen and Watson, Industrial Chemical Calculations, pp. 232-235, 321-325.

Transaction of American Institute of Chemical Engineers, Vol. 19, p. 173.

University of Wisconsin Engineering Experiment Station Bulletin No. 70.

Air, flowing through a pipe coil, is heated by radiation from furnace walls and by the convection currents of the hot gases.

Three options are described as to method of heating and other options may be supplied by using gases other than air.

(a) The pipe coil, through which air is flowing under pressure, is placed within the muffle of an electrically heated furnace.

(b) The pipe coil, through which air is flowing under pressure, is placed within the body of a pot assay furnace, or in the muffle of a gas-fired furnace.

(c) The pipe coil, through which air is flowing under pressure, is placed in the upper section of a specially constructed, gas-fired furnace.

Object. 1. To measure the quantity of heat, flowing under the chosen operation conditions.

2. To compare mathematically the various formulas available.

3. To select the formulas judged to be most applicable.

4. To resolve the total heat transferred into that transferred by convection, transferred by radiation, and that transferred by conduction.

5. To calculate heat balance and materials balance for the total operation.

Procedure. The operations required and the necessary equipment construction are nearly the same in all the options. This particular description applies to the pipe coil in the electrically heated muffle. The sample data and calculations given have been obtained by operating this equipment.

A rectangular coil of ½-inch seamless steel tubing approximately 16 feet long, is placed within the electrically heated muffle of the furnace. A special fire brick door is used to close the front of the muffle and in this are two holes for the passage of the coil inlet and outlet. Both pipes should be insulated for a few feet outside of the furnace.

Air, under pressure, is supplied at the inlet end of the pipe coil. The amount of air passing through the coil is computed from the cross section, the pressure and the velocity. The velocity is calculated from the reading of a pitot tube as taken on a differential gauge, making proper allowance for the Pitot tube constant and the specific gravity of the liquid in the gauge. The temperature of the pipe coil, near the entrance, near the middle and near the exit, is taken by using a chromel-alumel thermocouple and a proper reading instrument. The couples are installed in the tube wall at its mid point. The temperatures of the inlet air and of the exit air are taken similarly to that of the pipe coil, but for this purpose, couples of small diameter are used.

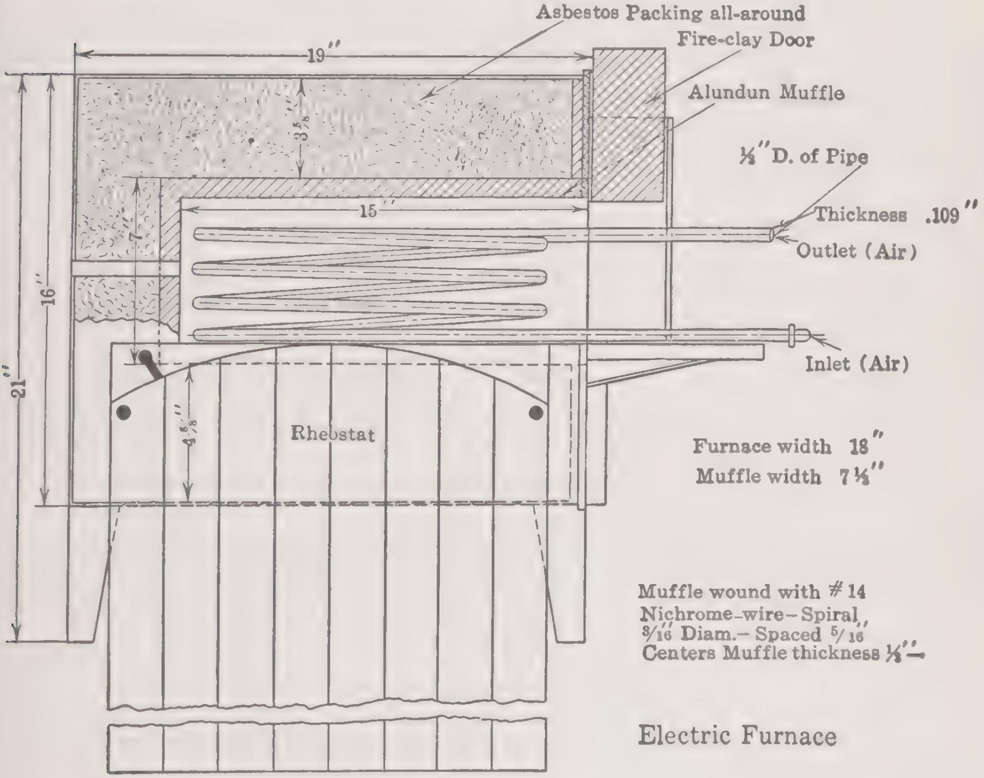

FIG. 1.—Electric furnace and pipe coil.

The temperature of the air within the muffle is taken periodically and at different positions by exploring with a movable aradiant thermocouple. The temperatures of the interior muffle walls are taken periodically by exploring with an optical or radiation pyrometer. The temperature of each of the exterior walls of the furnace is taken periodically, preferably with small thermocouples. The room temperature is also taken. A measurement of the energy consumed for heating is found in properly taken voltmeter and ammeter readings in the case of the electrically heated furnace, or from gas meter readings in the case of the gas heated furnaces. The humidity of the air is computed from the wet and dry bulb temperatures.

A furnace, from the operation of which the accompanying data was obtained, is shown in Sketch, Fig. 1.

The furnace is to be brought to between 600° and 900° C. and constant conditions obtained. The test is then run for one hour. Measure all input and output of electrical and heat energy and any conditions affecting them.

PRELIMINARY REPORT

Before writing the preliminary report inspect the furnace assigned, decide on the data to be obtained and the means for obtaining it. Consult the instructor, if in doubt as to the details of the equipment available.

The preliminary should be drafted along the following general lines:

Show the heat balance between heat supplied and heat expended in all possible ways.

Describe the detailed procedure of making the test and submit the data sheet considered to be satisfactory for this experiment.

Discuss possible limitations or inaccuracies in any of the measurements, such as:

1. Will a thermometer or pyrometer inserted in a tube to measure the temperature of a flowing gas, give the correct reading? If not, indicate how the correct value may be obtained.
2. How may a correction for cold junction error in the reading of the pyrometers be compensated?
3. Outline, giving exact details, a method which could be used to calibrate the Pitot tube. Discuss the limitatons and proper use of a Pitot tube.
4. What effect will the humidity of the air have on the measurements? How would humidity be measured?

State the method of calculating heat input, useful heat and efficiency.

State the data necessary to calculate the theoretical radiation loss and the film coefficients specified.

REPORT

Fill out the data sheets and record all indicated calculations (show the actual form of all calculations on other accompanying sheets).

Under "Outline of Test" state as briefly as possible, the procedure in such a way that it will be clear to an engineer unfamiliar with the particular experiment.

State any variations from the preliminary and the reasons.

Discuss any sources of error in the experiment.

State any possible improvements in the procedure.

SAMPLE DATA AND CALCULATIONS

Furnace dimensions:

Outside	1.54′	1.52′	1.33′
Inside	1.25′	0.625′	0.50′

Pipe in furnace:

Length	15.85′	
I.D.	9⁄32″,	0.0234′
O.D.	½″,	0.0416′

Cross sectional area 0.00043 ft.^{2}

Humidity:

Wet bulb temp. 85° F. $H = 0.014$ mols water vapor/mol of dry air (from chart, Fig. 16, p. 118, Hougen & Watson, Industrial Chemical Calculations.
Dry bulb temp. 65° F.

Temperatures in degrees F.:

Room	86	Outlet air	524
Furnace top	109	Furnace	788
Furnace front and back	131	Pipe at inlet	227
Furnace sides	100	Pipe at outlet	610
Inlet air	86		

Amperage, 21.75
Voltage, 80.0
Gauge pressure, 11 lbs./in.^{2}
Velocity head, 0.706 in. of water.
Calibration constant of pitot tube, 0.72.
Pipe containing pitot tube, ⅞″ standard pipe [1].
Pipe cross-sectional area, 0.304 in.^{2}, .00211 ft.^{2}
Barometer, 30.0 in. of Hg.
Time of test, 1 hr.

Input:

Electrical input = $EI/1000$ = kw.-hr.
Kw.-hr. × 3411 = B.t.u.
$EI \times 3.411$ = B.t.u. $= 21.75 \times 80.0 \times 3.411 = 5935.$

Output:

Useful heat

Density of air:

$$\left(\frac{11.0 + 14.6}{14.7}\right)\left(\frac{492}{546}\right)\left(\frac{29}{359}\right) = 0.1263 \text{ lbs./ft.}^3$$

There is 0.014 mol of water vapor/mol of dry air.

---

[1] It is to be noted that the pipe, in which the pilot tube is located, is ⅞″ standard pipe while the coil in the furnace is ½″.

Density of water vapor:

$$(25.6/14.7) \quad (492/546) \quad (0.014 \times 18)/359 = 0.0011 \text{ lb./ft.}^3$$

The density of the humid air:

$$0.1263 + 0.0011 = 0.1275 \text{ lb./ft.}^3$$

The velocity head, 0.706 in. of water.
The density of water is 62.42 lbs./ft.^{3}
The velocity head:

$$62.42/0.1274 \times 0.706/12 = 28.8 \text{ ft. of air.}$$

The velocity:

$$V(\text{ft./sec.}) = 0.72\sqrt{2\,g \times 28.8}$$

$$V = 0.72\sqrt{64.4 \times 28.8} = 31.01 \text{ ft./sec.}$$

The volume of air passing the pitot tube per hour:
(Cross-sectional area of inlet pipe × V × sec./hr.)

$$0.00211 \times 31.01 \times 3600 = 235.45 \text{ ft.}^3$$

The weight of air per hour:

$$235.45 \times 0.1263 = 29.74 \text{ lbs. or } 29.74/29 = 1.03 \text{ mols.}$$

Sensible heat in the dry air:

Mean molal heat capacity of O_2 and N_2 between 86° F. and 524°F. = 7.05. (*ibid*, Fig. 29, p. 194)

Therefore, the sensible heat:

$$7.05(524\text{–}86) = 3084 \text{ B.t.u./lb. mol.}$$

$$3084 \times 1.03 = 3176 \text{ B.t.u.}$$

Sensible heat in water vapor:

Mean molal heat capacity of water vapor between 86° F and 524° F = 8.53 (ibid, loc. cit.)

Therefore, the sensible heat:

$$8.53(524\text{–}86.0)0.0144 = 54.9 \text{ B.t.u.}$$

The total useful heat:

$$54.9 + 3176 = 3231 \text{ B.t.u.}$$

The efficiency of the furnace:

$$3231/5935 \times 100 = 54.5\%$$

Heat losses:

Radiation losses

$$q = 0.172(A) \quad [(T_1/100)^4 - (T_2/100)^4] \quad (F_A) \quad (F_E)$$

A (front and back) $= 2 \times 1.52 \times 1.33 = 4.05 \text{ ft.}^2$

$F_A = 1$

$F_E = 0.9$

Heat lost from front and back:

$$T_1 = 460 + 131 = 591$$
$$T_2 = 460 + 86 = 546$$
$$q = 4.05 \times 0.172 \times 0.9\,(5.91^4 - 5.46^4)$$
$$q = 0.625 \times 331.3 = 209 \text{ B.t.u./hr.}$$

Heat lost from sides:

$$A \text{ (sides} = 2 \times 1.54 \times 1.33 = 4.1 \text{ ft.}^2$$
$$T_1 = 460 + 100 = 560$$
$$T_2 = 460 + 86 = 546$$
$$q = 4.1 \times 0.172 \times 0.9(5.6^4 - 5.46^4)$$
$$q = 0.635 \times 101.8 = 65 \text{ B.t.u./hr.}$$

Heat lost from top:

$$A \text{ (top)} = 1.54 \times 1.52 = 2.34 \text{ ft.}^2$$
$$T_1 = 460 + 109 = 569$$
$$T_2 = 460 + 86 = 546$$
$$q = 2.34 \times 0.172 \times 0.9\,(5.69^4 - 5.464^4)$$
$$q = 0.362 \times 157 = 57 \text{ B.t.u.}$$

Total heat lost by radiation:

$$209 + 65 + 57 = 331 \text{ B.t.u./hr.}$$
$$331/5935 \times 100 = 5.6\% \text{ of heat input}$$

Heat lost by convection:

For sides, front and back; $h_c = 0.3\ \Delta t 0.25$ (McAdams, eq. 29, p. 244)
For top; $h_c = 0.38\ \Delta_t 0.25$ (ibid, eq. 30, p. 245)

Sides:

$$\Delta_t = 100 - 86 = 14$$
$$h_c = 0.3 \times 14^{0.25} = 1.934$$
$$A = 4.1 \text{ ft.}^2$$
$$\text{B.t.u.} = 4.1 \times 1.934 \times 14 = 112$$

Front and back:

$$\Delta_t = 131 - 86 = 45$$
$$h_c = 0.3 \times 45^{0.25} = 2.6$$
$$A = 4.05 \text{ ft.}^2$$
$$\text{B.t.u.} = 4.05 \times 2.6 \times 45 = 470$$

Top:

$$\Delta_t = 109 - 86 = 23$$
$$h_c = 0.38 \times 23^{0.25} = 2.2$$
$$A = 2.34 \text{ ft.}^2$$
$$\text{B.t.u.} = 2.34 \times 2.2 \times 23 = 117$$

Total B.t.u. lost by convection:

$$112 + 470 + 117 = 699$$
$$699/5935 \times 100 = 11.8\% \text{ of input energy}$$

Heat Balance:

Heat input	Heat output	
100%	Useful heat................	54.5%
	Radiation loss.............	5.6%
	Convection loss............	11.8%
	Accounted for..............	71.9%
	Unaccounted................	28.1%

Among the unaccounted losses are:
- Heat loss through the bottom of the furnace
- Heat lost through cracks (front and back)
- Heat lost from the ends of the pipe

Theoretical Consideration of Heat Transfer:

Heat transferred to the pipe by radiation:

$$q = 0.172 \times A\,[(T_1/100)^4 - (T_2/100)^4]\,F_A F_E$$
$$F_A = 1$$
$$F_e = 0.736 \text{ (oxidized iron) (Table I, p. 46, McAdams)}$$

The area of the pipe is $15.85 \times 0.0416 \times 3.1416 = 2.07$ ft.^{2}
Due to the screening effect of one bend of the coil on another, the area exposed to radiation is $2.07/2 = 1.035 = A$

$$T_1 = 788 + 460 = 1248$$
$$T_2 = (227 + 610)/2 + 460 = 879$$
$$q = 0.736 \times 0.172 \times 1.035\,(12.48^4 - 8.79^4)$$
$$q = 0.736 \times 0.172 \times 1.035 \times 18{,}290 = 2396 \text{ B.t.u./hr.}$$

Heat transferred to the pipe by convection:

$$h_c = 0.42 \times (\Delta t/D')^{0.25},\ D',\ \text{O.D. of pipe in inches (McAdams, eq. 27, p. 244)}$$
$$\Delta t = 788 - 369 = 419$$
$$D' = \tfrac{1}{2}''$$
$$h_c = 0.42 \times (419 \times 2)^{0.25} = 2.26$$
$$\text{B.t.u.} = 2.26 \times 419 \times 3.1416 \times 0.0416 \times 15.85 = 1788.8$$

B.t.u. transferred by radiation and convection:

$$1788.8 + 2396 = 4184.8$$

EXPERIMENT NO. 17

Flow of Heat

REFERENCES

A.I.T. student reports, Experiments 13 and 14.

Barr and Berger, A Simple, Portable Aradiant Thermocouple, Journal of Industrial and Engineering Chemistry, Analytical Edition, Vol. 8, p. 393 (1936).

Industrial Heat Transfer, Schack, pp. 128-129.

Industrial Heat Transfer, Schack, pp. 175-179.

Walker, Lewis, McAdams, and Gilliland, Principles of Chemical Engineering.

Badger and McCabe, Elements of Chemical Engineering.

McAdams, Heat Transmission, pp. 56-57 and 79 seq.

Haslam and Russell, Fuels and Their Combustion, pp. 193-227.

Water flowing through a pipe coil is heated by radiation and convection in a gas-fired furnace.

Two methods of operation are offered:

(a) The pipe coil is placed in a gas-fired furnace, such as a pot furnace.

(b) The pipe coil is placed in the combustion chamber of a specially designed gas-heated furnace where the gas is burned in a radiant unit along each side.

The description given is for option (b); the sample data and calculations are given for both options.

Water flowing through the pipe coil may be replaced by any other liquid, the heat transfer characteristics of which it may be desired to study.

This experiment affords opportunity for the experimental study of most of the conditions under which heat may be transferred.

Some of the conditions under which transfer is occurring have not been studied with sufficient care to enable one to select formulae for film coefficients with unerring accuracy. Judgment and discretion are required in choosing the formulae which seem most nearly applicable.

There are also some complications in the measurement of the temperature of the furnace gases, with a thermocouple, in the zone heated primarily by radiation, and in measuring the temperature and the velocity of the air passing through the pipe coil in the upper section of the furnace.

Considerable information is to be found in recent technical literature and it is recommended that a search, covering the years 1934 to date, be made during the preparation of the preliminary report.

Object. To study various factors influencing flow of heat by conduction, by convection, and by radiation.

To compare thermal conductivity, and corresponding heat losses, of varying types of furnace walls.

To determine rate of heat flow from furnace chamber to water flowing in a pipe coil; expressed as total heat and as B.t.u. per sq. ft. per hour per degree temperature difference.

To determine experimental film coefficients and comparison between these and the calculated coefficients.

To resolve sources of heat into: (a) convection, (b) radiation.

To resolve heat utilization into: (a) conduction, (b) convection, (c) radiation.

To calculate: Sensible heat in flue gas; heat balance for entire operation; cost of heating 1000 gals. of water to 210° F., gas costing 70 cents per 1000 cu. ft. and operating conditions as in this experiment.

Gas-fired Furnace

Explanation of Illustrations

A. Water coil — extra heavy cold drawn steel pipe in ½ inch standard iron pipe size
B. Thermometer well
C. Water control valve
D. Air control valve
E. Air coil — standard ¾ inch wrought iron pipe
F. Nonpareil insulating brick, 2½″ × 4½″ × 9″ — Armstrong Cork & Insulation Company
G. 85% magnesia block, 3″ × 18″ × 2″ — Johns-Manville, Inc.
H. Fire brick refractory, 1½ inch thickness
I. Entrance hole for aradiant convection pyrometer. Gas temperature determination
J. Radiant fire unit, 8″ × 14″
K. Pitot tube for air velocity and pressure measurement
L. Gas sampler and cooler
M. Angle irons, 2″ × 2″ × ⅛″
N. Supports for furnace — iron pipe, 5″ diameter
O. Silica block lining of furnace, 3″ thickness
P. Gas burner valve
Q. Improved Asbestocel, 4 ply per inch, 2″ thickness — Johns-Manville

The entire furnace has been covered with a layer of heavy cardboard, 3⁄16 inch thick.

The numerals on the diagrams, Figs. 2, 3 and 4, indicate thermocouple locations and therefore points at which operating temperatures are taken.

Equipment. The furnace to be used is one designed and built for this experiment. Its construction, arrangement and dimensions are detailed in the accompanying diagrams.

The heated section consists of a chamber with side walls built of refractory shapes in which city gas is burned, they are, in fact, two "radiant fire" units as customarily used in a gas grate. The pipe coil through which the water flows lies between these heating surfaces. Thermocouples are located at such points as will indicate the pipe coil temperature, the heating chamber temperature, the temperature of various parts of the furnace walls, and the temperature of the stack gases. A peep hole is provided through which the temperature of the radiant brick work can be taken by the use of a radiation pyrometer.

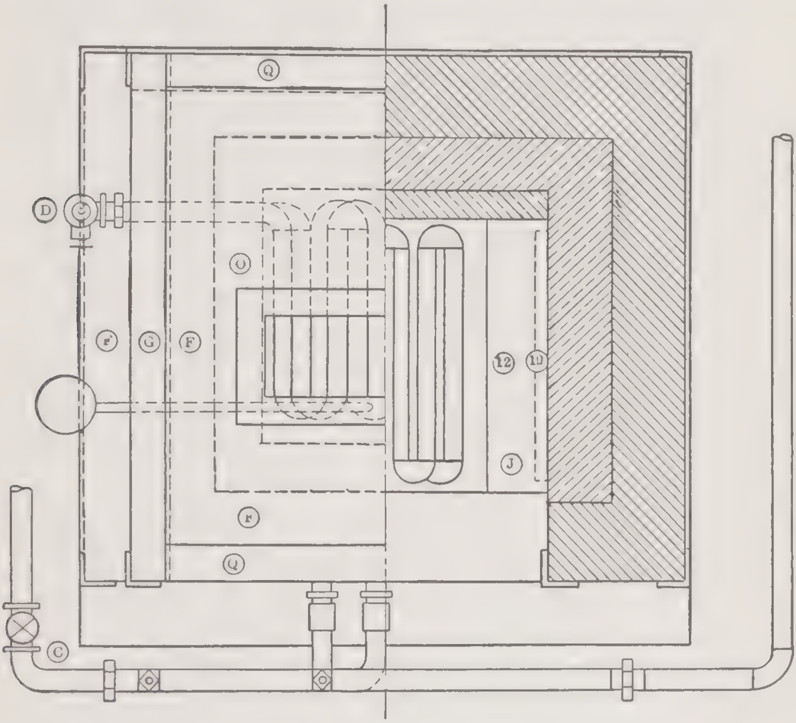

Fig. 2.—Furnace details.

The temperature of the incoming and outflowing water is taken by thermocouples.

The quantity of water flowing through the pipe coil during the test is weighed.

Gas consumed during the test is measured by a gas meter.

Stack gas samples are taken in a metal gas sample container, first drawing the hot gas through a water-cooled copper coil. Analysis of the stack gases are made, using any of the standard equipment, and extreme care.

Furnace construction and dimensions are detailed on the accompanying

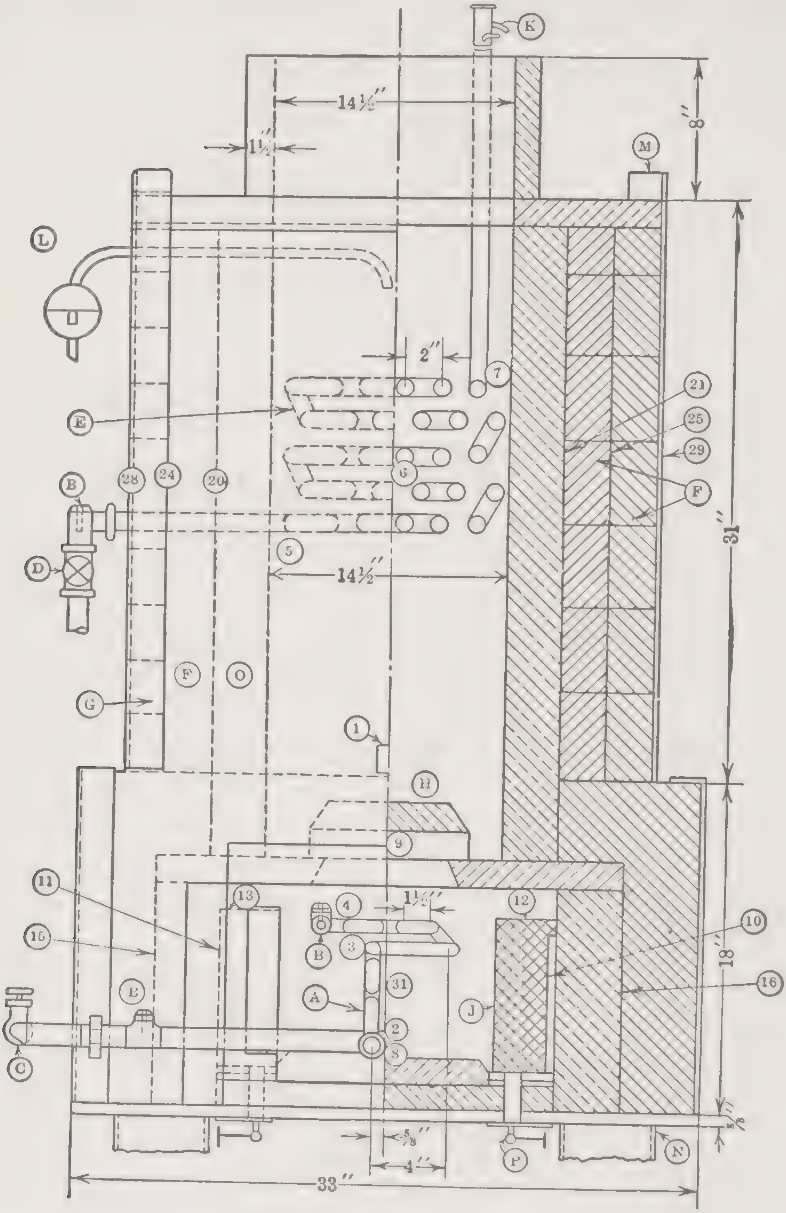

Fig. 3.—Furnace details.

diagrams and such data as may be necessary for the calculations requested can be read from the diagrams. Such other data should be taken as it is deemed necessary for the calculations requested.

The locations of the various thermocouples are tabulated on page 133.

Procedure. The experiment is commenced by starting the flow of water through the pipe coil at such a rate as is believed to be satisfactory (five to ten pounds per minute). The gas burners are then lighted. The heating of the furnace chamber and the flow of the water through the pipe coil are then continued until there is little if any, variation in the temperature of the outflowing water and in the temperature of the furnace

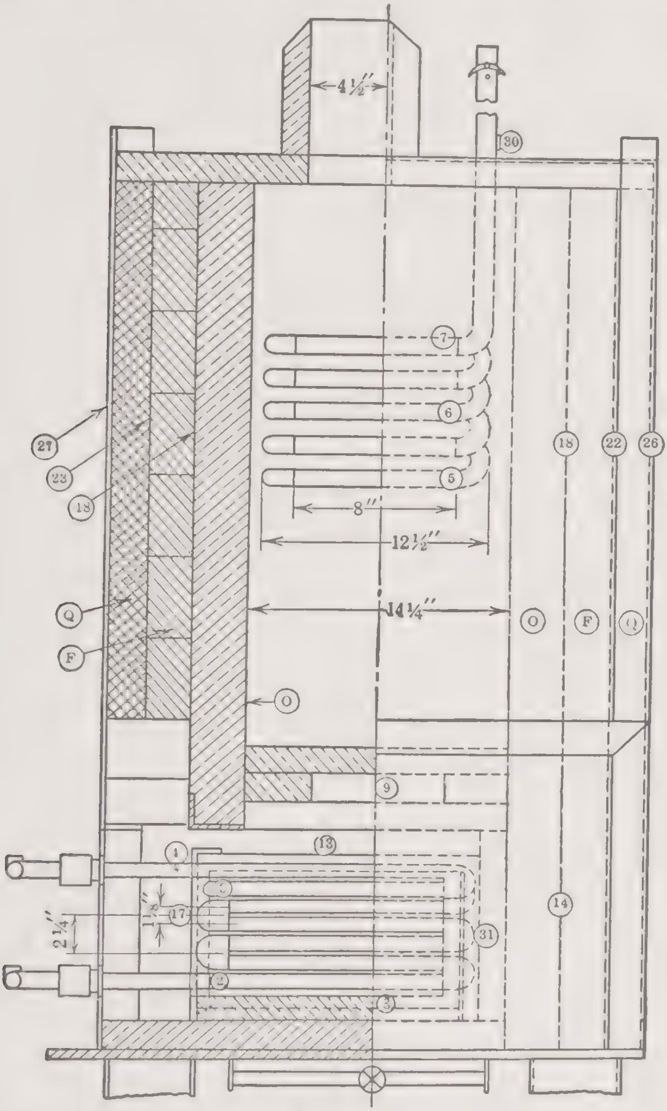

Fig. 4.—Furnace details.

chamber. The taking of data is commenced. The experiment should be continued for at least one hour after these constant conditions of operation have been reached.

The temperature of the radiant fire units is determined by means of a total radiation pyrometer. The gas temperatures in the water coil area, below the air coil, and above the air coil are measured by means of the aradiant convection pyrometer inserted through the openings provided.[1] The temperature above and below the air coil can be taken in one position each, but the water coil area must be covered by at least a 6 point traverse taking readings adjacent to various sections of the pipe coil. It is not possible to accurately determine these gas temperatures, and consequently, the outside gas film coefficients without the use of this pyrometer. The error of a common thermocouple used in this furnace varies from −100° F. to +300° F., due to radiation to and from the couple. In the operation of this pyrometer a laboratory vacuum pump is used to draw the gas through the pyrometer.

The vacuum pump is also used to draw the gas sample through the sampler on the upper left side of the furnace. A metal gas sample bottle is connected in series between the vacuum pump and the sampler. The gas sample bottle is opened and the pump is run until the bottle begins to get warm, when the pump is stopped, and both ends of the sample bottle are closed. Care in making the gas analysis is again emphasized. The most accurate methods and the best technique are required. Slight errors are magnified in the calculations arising from these gas analyses.

The test run should be for one hour and readings are made at ten minute intervals. Make no changes in rates of water, air, or gas flow during the run. A run in which rates and temperatures have not been reasonably constant must be repeated.

In shutting down the furnace, the first step is to turn off the gas to both burners. After ten minutes, turn off air and water. At no time should the gas be burning without air and water flowing in the coils. Operations under such conditions will burn out the pipe coils and ruin the furnace.

Promptly report any thermocouples which do not give readings or the failure of any other part of the furnace, so that it may be repaired before the furnace is desired for further experimental work.

PRELIMINARY REPORT

Discuss, briefly but concisely, the conditions under which heat transfer is occurring in this experiment. Trace each transfer completely. Cite the various equations which may be used in calculating the theoretical transfer.

---

[1] See Measurement of Temperature, page 62.

Mention any operating conditions in this experiment which deviate from the conditions under which the theoretical equations are applicable.

In case there are such deviations, discuss their probable effect.

Set up a detailed heat balance for the experiment, listing each source of heat and all the ways in which this heat is utilized or dissipated. Heating value of the gas used is to be calculated from its analysis or preferably determined directly using the gas calorimeter.

Where two streams of material converge and only one can be measured, it is possible to calculate the other by reference to a common element on the basis of analysis before and after mixing. Discuss the application of this method for the calculation of the unmeasurable volumes of air and flue gas.

REPORT

Supply the figures necessary to complete the heat balance set up in the preliminary report. Show the calculation of this heat balance on other sheets of the report.

Compare the experimental figures with the theoretically calculated ones on each item where such comparison is possible. Attempt to explain any discrepancies between experimental figures and calculated ones.

Set up a materials balance analogous to the heat balance.

Discuss the efficiency of combustion attained, as measured by the analysis of the escaping products of combustion.

Suggest other formulas than those used for the calculation of film coefficients on both coils.

Compare the amounts of heat supplied by radiation and by convection in each heating section.

Compare the flow of heat through the various types of insulation used in the furnace walls.

What effect has humidity on the heat balance?

State any variations from the preliminary and reasons for such variations.

Discuss various sources of error in the experiment.

State any possible improvements in the procedure.

OPERATING INSTRUCTIONS FOR GAS-FIRED FURNACE, *Option C*

Previous procedure, on page 118, was for the operation of the special gas-fired furnace when only a study was to be made of the heat transfer to water in the pipe coil. The instructions given herewith cover the operation of the furnace when a study is being made of the transfer of heat to water in the pipe coil in the lower section of the furnace and also to air flowing in the pipe coil in the upper section of the furnace.

The purpose of the operation of this furnace is the experimental determination of rates of heat transfer, under furnace conditions, to air and water flowing in pipe coils. The heat transfer coefficients experimentally determined are compared with values calculated by the use of various formulas found in the literature. A heat balance is also determined for the entire piece of apparatus, and heat losses through walls and in flue gases are measured.

It is most essential, in the performance of this experiment, that temperatures be taken at all points provided and that all rates of material flow be measured.

The first operating details are to assure a flow of water in the lower coil and a flow of air in the upper coil by opening the proper valves.

It is essential that the discharge of the water line be at a point above the level of the water coil to be certain that the coil is completely filled. It has been found convenient to discharge the water from the coil into a small tank which then drains in the weighing tanks.

The flow of air through the air coil is checked by noting the reading on the inclined draft gauge which connects with the Pitot tube in the air discharge line.

When sure that both water and air are flowing through the appropriate coils, light the gas at the radiant fire units on either side of the water coil area. The furnace, due to its heavy wall construction, heats slowly. It, therefore, should be operated for at least two hours before readings of any kind are taken. This will assure fairly constant operating conditions. A 0-100° C. thermometer should be placed in the hole in the top of the switch box for the determination of the cold junction temperature. Since the cold junction is at the switch box it is necessary to use the compensator on the potentiometer. This is the small dial on the left. Its reading (using the correct scale as shown by the position of the plug above it) should be set to correspond with the cold junction millivoltage as given on a chart of cold junction temperatures versus millivoltage. The potentiometer readings will then be in terms of true temperature.

The rate of air flow in the upper pipe coil is determined by the inclined tube draft gauge and Pitot tube. The static pressure is measured on the vertical U-tube to the right of the switch box. Readings of the inclined tube draft gauge are multiplied by 0.9 to give inches of water. The vertical tube contains water.

The rate of water flow is determined by weighing the water in barrels below the anti-siphon tank. The rate should be such that the temperature rises at least 10° C. A weighing period should not begin within 5 minutes of a water rate change, since it takes time for the water level in the anti-siphon to adjust itself.

A typical listing of thermocouple locations on the equipment is appended.

GAS-FIRED FURNACE

Thermocouple Locations (EXPERIMENTS NO. 17 AND NO. 18)

1. Aradiant convection pyrometer
2. Beginning of water coil
3. Center of water coil
4. Outlet of water coil
5. Beginning of air coil
6. Center of air coil
7. Outlet “ “ “
8. Surface, floor of water coil area
9. Surface, above water coil area
10. Behind right hand radiant unit
11. “ left “ “ “
12. Above right “ “ “
13. “ left “ “ “
14. Back, lower section, between silica lining and Nonpareil brick
15. Left, “ “ “ “ “ “ “ “
16. Right, “ “ “ “ “ “ “ “
17. Front, “ “ inside surface of door
18. Back, top section, between silica lining and Nonpareil brick
19. Front, “ “ “ “ “ “ “ “
20. Left, “ “ “ “ “ “ “ “
21. Right, “ “ “ “ “ “ “ “
22. Back top section, between Nonpareil and Asbestocel
23. Front, “ “ “ “ “ “
24. Left, “ “ “ “ “ 85% magnesia block
25. Right, “ “ “ “ “ outer Nonpareil brick
26. Back, “ “ outside surface of Asbestocel
27. Front, “ “ “ “ “ “
28. Left, “ “ “ “ “ 85% magnesia block
29. Right “ “ surface of outer Nonpareil brick
30. Outlet air temperature, built-in aradiant convection pyrometer
31. Back, inside surface, silica lining of water coil area

SAMPLE DATA AND CALCULATIONS, *Option C*

(Data and Calculations by Berger)

(Number designates thermocouple)

The top box points are the air coil.

	°F
1. Outside connection.	
2. Temperature at beginning of water coil	82
3. Center of water coil	108
4. Outlet of water coil	126
5. 6. No connection 7.	
8. Surface, floor of water coil area	720
9. Surface, roof of water coil area	833
10. Behind right radiant unit	645
11. Behind left “ “	466
12. Above right “ “	600
13. Above left “ “	871
14. Back, lower section, between silica and Nonpareil (dead)	243
15. Left, “ “ “ “ “ “	328
16. Right, “ “ “ “ “ “	259
17. Front, “ “ inside door surface	650
18. Back, top section, between silica and Nonpareil	344
19. Front, “ “ “ “ “ “	361
20. Left, “ “ “ “ “ “ (dead)	386
21. Right, “ “ “ “ “ “	364
22. Back, “ “ “ Nonpareil and Asbestocel	132
23. Front, “ “ “ “ “ “	156
24. Left, “ “ “ “ “ magnesia	139
25. Right, “ “ “ “ “ Nonpareil	135
26. Back, “ “ outside Asbestocel	75
27. Front, “ “ “ “	84
28. Left, “ “ “ magnesia block	77
29. Right, “ “ “ Nonpareil	84
30. No connection.	
31. Back, inside surface, water coil area	795
33. No connection.	
Wet bulb temperature	51
Dry bulb temperature	70
Inlet water temperature	47
Outlet water temperature	113
Inlet air temperature	74.5
Outlet air temperature	541
Radiant unit temperature	1500
Temperature above air coil	670
Temperature below air coil	960
Temperature of room	70

Gas consumption	99 cu. ft.
Air static pressure	0.01 in. water
Velocity head	0.33 in., 0.9 sp.gr.
Water used per hour	372 pounds

SUMMARY OF CALCULATED DATA

(Data obtained and calculated by Berger)

Water Coil

Total heat transfer	24,552	B.t.u./hr.
Overall coefficient	10.51	B.t.u./hr./sq.ft./° F.
Water film, experimental	304	“ “ “ “
Water film, theoretical	266	“ “ “ “
Gas film, theoretical, horizontal section	2.01	“ “ “ “
Gas film, theoretical, vertical section	1.80	“ “ “ “
Gas film, experimental	0.693	“ “ “ “
Radiation transfer	22,742	B.t.u./hr.
Convection transfer	1,810	“ “

Air Coil

Total heat transfer	2,200	B.t.u./hr.
Overall coefficient	0.578	B.t.u./hr./sq.ft./° F.
Air film, experimental	0.802	“ “ “ “
Air film, theoretical	6.30	“ “ “ “
Gas film, experimental	2.91	“ “ “ “
Gas film, theoretical	2.13	“ “ “ “

Heat Balance of Entire Process

Input

1. Heating value of fuel gas	74,840	B.t.u.
2. Sensible heat of fuel gas	0	“
3. Sensible heat of dry air	0	“
4. Heat content of water vapor in air	367	“
5. Heat content of piped water	−8,556	“
6. Heat content of piped air	71	“
Total input	66,722	“

Output

1. Heating value of flue gas	0	B.t.u.
2. Heat content of dry flue gas	20,030	“
3. Heat content of water vapor in flue gas	10,150	“
4. Heat content of piped water	15,996	“
5. Heat content of piped air	2,271	“
6. Accounted for wall losses, etc	8,637	“
7. Unaccounted for and other losses	9,638	“
Total output	66,722	“

Flow of Heat from Furnace to Water Flowing in Pipes:

B.t.u. to water:

Weight of water per hour	372 pounds
Entering temperature	47° F.
Exit temperature	113° F.
Heat transfer 372 (113–47)	24,552 B.t.u./hr.
Mean area of water film	2.840 sq. ft.
Log mean water temperature	75.3° F.
Mean furnace temperature	775° F.

The mean furnace temperature given above was determined by a 14 point traverse, of the area surrounding the water pipe coil, with the aradiant convection pyrometer.

Temperature difference from furnace to water . . . . 700° F.

Apparent overall coefficient for entire transfer

$$U = \frac{24{,}552}{3.341 \times 700} = 10.51 \text{ B.t.u./hr./sq. ft./° F.}$$

Mean temperature at center of metal pipe wall . . . . 105° F.

Determination of drop through wall:

$$q = \frac{kAdt}{1}$$

$A = 3.341$ sq. ft.
$dt = ?$
$l = 0.0091$ ft.
$k = 26$ B.t.u./hr./sq. ft./ft./° F.
$q = 24{,}552$ B.t.u./hr.

$$dt = 2.6° \text{ F.}$$

Temperature on water side	103.7° F.
Drop through water film (103.7 − 75.3)	28.4° F.

$$h_w = \frac{24{,}552}{2.840 \times 28.4} = 304 \text{ B.t.u./hr./sq. ft./° F.}$$

Theoretical Water Film Coefficient:

$$\frac{hD}{k} = 0.0225 \left(\frac{DG}{u}\right)^{.8} \left(\frac{C_p u}{k}\right)^{.4}$$

h = film coefficient, B.t.u./hr./sq. ft./° F.
D = inside pipe diameter, feet −0.052
C_p = sp. heat of film material at film temp. . . . . 1.0 B.t.u./lb./° F.
k = thermal conductivity of film material at mean film temperature 0.351
u = viscosity at mean film temperature . . . . 1.849 lb./hr./ft.
G = mass velocity . . . . 175,000 lb./hr./sq. ft.

$$h_w = 266 \text{ B.t.u./hr./sq. ft./° F.}$$

Apparent outside gas film coefficient:

$$h_g = \frac{24{,}552}{3.921 \times 667} = 9.29 \text{ B.t.u./hr./sq. ft./° F.}$$

However, this coefficient has no real meaning since such a large proportion of the heat is transferred by radiation, and the proportions of heat transferred in each section of the coil are not known.

Theoretical Outside Gas Film Coefficients:

Horizontal section of pipe coil

$$h_g = \frac{1.60T^{\cdot 3}G^{2/3}}{D^{1/3}}$$

Mean gas temperature	825° F. 12850° F.
G mass velocity	0.0515 lb./sec./sq. ft.
D outside diameter	0.840 inch

$$h_g = 2.01 \text{ B.t.u./hr./sq. ft./° F.}$$

Vertical section of pipe coil using Fig. 14, p. 862, Perry, Chemical Engineer's Handbook.

p = 1 atmosphere
t_m = 308° C. since mean gas temperature = 650° F.
D = 0.840 inch
t_f = 190° C.
h_g = 1.80 B.t.u./hr./sq. ft./° F.

Radiation Transfer to Water Coil:

From radiant fire units to water coil:

$$q = 0.172A \left(\frac{T_1}{100}\right)^4 - \left(\frac{T_2}{100}\right)^4 F_A F_E$$

T_1 = 1959° R.
T_2 = 565° R.
F_A = 1
F_E = 0.79

Factor of comparison from Fig. 5, p. 890, Perry = 0.86

A = 1.224 sq. ft.

$$q = (0.172)\ (1.224) \left(\frac{1959}{100}\right)^4 - \left(\frac{565}{100}\right)^4 (1)\ (0.79)\ (0.86) = 21{,}000 \text{ B.t.u./hr.}$$

From water coil area floor to coil:

T_1 = 1180° R.
T_2 = 565° R.
F_A = 0.65
F_E = 0.79
A = 0.524 sq. ft.

Comparison factor—0.68

$$q = 580 \text{ B.t.u./hr.}$$

From roof of water coil area to coil:

$$
\begin{aligned}
T_1 &= 1293^\circ \text{ R.}\\
T_2 &= 565^\circ \text{ R.}\\
F_A &= 0.65\\
F_E &= 0.79\\
A &= 0.524 \text{ sq. ft.}
\end{aligned}
$$

Comparison factor—0.68

$$q = 825 \text{ B.t.u./hr.}$$

From back wall of water coil area to coil:

$$
\begin{aligned}
T_1 &= 1265^\circ \text{ R.}\\
T_2 &= 565^\circ \text{ R.}\\
F_A &= 0.65\\
F_E &= 0.79\\
A &= 0.10 \text{ sq. ft.}\\
q &= 210 \text{ B.t.u./hr.}
\end{aligned}
$$

From front wall of water coil area to coil:

$$
\begin{aligned}
T_1 &= 1110^\circ \text{ R.}\\
T_2 &= 565^\circ \text{ R.}\\
F_A &= 0.65\\
F_E &= 0.79\\
A &= 0.10 \text{ sq. ft.}\\
q &= 127 \text{ B.t.u./hr.}
\end{aligned}
$$

Total radiation transfer	22,742 B.t.u./hr.
Which leaves by convection	1810 B.t.u./hr.

or convection film coefficient:

$$\frac{1810}{3.921 \times 667} = 0.693 \text{ B.t.u./hr./sq. ft./}^\circ \text{ F.}$$

Flow of Heat from Furnace to Air Flowing in Pipe Coil:

Outside temperature at bottom of air coil	690° F.
Outside temperature at top of air coil	670° F.

Air pipe temperatures	
Inlet	568° F.
Center	645° F.
Outlet	602° F.

Air temperatures	
In	74.5° F.
Out	540.0° F.

Pitot tube reading	0.315 in. water
Barometer	74.335 cm. Hg
Static pressure	0.009 in. water
Wet bulb temperature	51° F.
Dry bulb temperature	70° F.
Humidity of air	0.004 molal

Unit volume of air at pitot tube:

$$13.75 \times 760/743 \times 1000/530 = 26.5 \text{ cu. ft./lb. dry air or } 0.0376 \text{ lb./cu. ft.}$$

and wet air:

$$0.0376 \quad 0.0376/29 \times 0.004 \times 18 = 0.0377 \text{ lb. wet air per cu. ft. at } 540° \text{ F.}$$

Maximum velocity from pitot tube reading	52.8 ft./sec.
Factor of pitot tube from Perry (p. 691)	0.752

$$\text{Average velocity} = 52.8 \times 0.752 = 39.7 \text{ ft./sec.}$$

$$\text{Cross-section of pipe} = 0.00369 \text{ sq. ft.}$$

Volume of air per hour:

$$39.7 \times 0.00369 \times 3600 = 514 \text{ cu. ft./hr.}$$
$$\text{or } 514 \times 0.0377 = 19.3 \text{ lb./hr.}$$

Total transfer:

$$(540 - 74.5)\ (19.3)\ (0.24) \quad 19{,}330 - (18 \times 44) \quad (8.3 \times 509.5)$$
$$0.00267 = 2200 \text{ B.t.u./hr.}$$

Average pipe temperature	603° F.
Log mean air temperature	235° F.
Temperature drop	368° F.

Experimental air film coefficient:

$$\frac{2200}{368 \times 7.466} = 0.802 \text{ B.t.u./hr./sq. ft./° F.}$$

Theoretical Air Film Coefficient:

$$\frac{hD}{k} = 0.024 \left(\frac{DG}{u}\right)^{.8} \left(\frac{C_p u}{k}\right)^{.4}$$

h = film coefficient	B.t.u./hr./sq. ft./° F.
D = pipe diameter	0.0687 ft.
k = thermal conductivity of film at mean film temperature	0.0201
u = viscosity of film material at mean film temperature	0.0654 lb./hr./ft.
G = mass velocity	5210 lb./hr./sq. ft.
C_p = specific heat of film material	0.245 B.t.u./lb. ° F.

$$h_a = 6.30 \text{ B.t.u./hr./sq. ft./° F.}$$

Overall air pipe coefficient:

$$U = \frac{2200}{8.555 \times 445} = 0.578 \text{ B.t.u./hr./sq. ft./° F.}$$

Experimental outside gas film coefficient:

$$h_g = \frac{2200}{9.778 \times 77} = 2.91 \text{ B.t.u./hr./sq. ft./° F.}$$

Theoretical outside gas film coefficient:

$$h_g = \frac{1.60T^{0.3}\,G^{2/3}}{D^{1/3}} \text{ (Ind. Eng. Chem., } \mathit{24}\text{, 505 (1932)}$$

T = absolute gas temperature	1140° R
G = mass velocity	0.0665 lb./sec./sq. ft.
D = pipe diameter, inches	1.050

$$h_g = 2.13 \text{ B.t.u./hr./sq. ft./° F.}$$

Heat Losses through Walls:

1. Upper section, front wall:

All calculations are based on temperature drop across inner layer of Armstrong nonpareil insulating brick. Conductivities of brick at various mean brick temperatures are given on the accompanying graph (Fig. 5).

Temperature on inside surface, #19	361° F.
Temperature on outside surface, #23	156° F.
Inner area	4.06 sq. ft.
Outer area	5.15 " "
Log mean area	4.60 " "
Thickness	2.50 in.
Mean temperature of brick	259° F.
Conductivity at this temperature	0.80 B.t.u./sq. ft./hr./°F./in.

Loss = 302 B.t.u./hr.

2. Upper section, rear wall:

Temperature on inside surface, #18	344° F.
Temperature on outside surface, #22	132° F.
Inner area	4.06 sq. ft
Outer area	5.15 " "
Log mean area	4.60 " "
Thickness	2.50 in.
Mean temperature of brick	238° F.
Conductivity at this temperature	0.79

Loss = 308 B.t.u./hr.

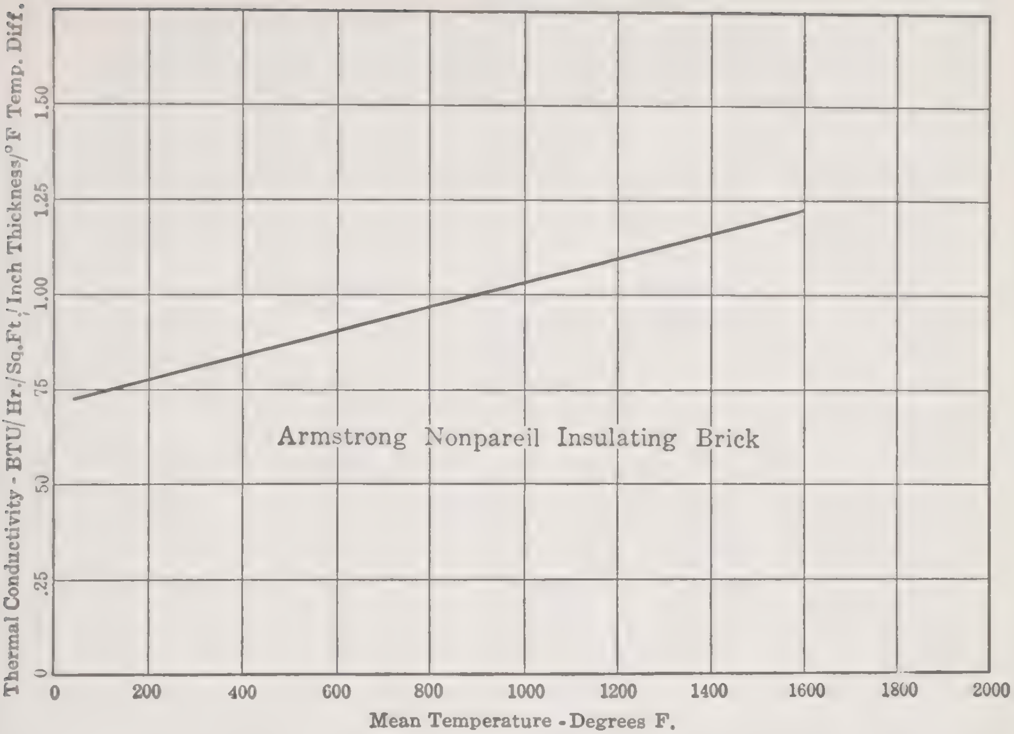

Fig. 5.—Thermal conductivity, Armstrong brick.

3. Upper section, left wall:

Temperature on inside surface, #20	386° F.
Temperature on outside surface, #24	139° F.
Inner area	4.48 sq. ft.
Outer area	5.57 " "
Log mean area	5.07 " "
Thickness	2.50 in.
Mean temperature of brick	263° F.
Conductivity at this temperature	0.80

Loss = 401 B.t.u./hr.

4. Upper section, right wall:

Temperature on inside surface, #21	364° F.
Temperature on outside surface, #25	135° F.
Inner area	4.48 sq. ft.
Outer area	5.57 " "
Log mean area	5.07 " "
Thickness	2.50 in.
Mean brick temperature	250° F.
Conductivity at this temperature	0.795

Loss = 369 B.t.u./hr.

5. Lower section, back wall:

Basis: Drop across nonpareil outer layer

Temperature on inside surface, # 14	243° F.
Temperature on outside surface, air	75° F.
Inner area	3.15 sq. ft.
Outer area	4.28 “ “
Log mean area	3.68 “ “
Thickness	4.50 in.
Mean brick temperature	159° F.
Conductivity at this temperature	0.76

Loss = 105 B.t.u./hr.

6. Lower section, left wall:

Temperature on inside surface, # 15	328° F.
Temperature on outside surface, air	75° F.
Inner area	2.77 sq. ft.
Outer area	3.69 “ “
Log mean area	3.21 “ “
Thickness	4.50 in.
Mean brick temperature	202° F.
Conductivity at this temperature	0.775

Loss = 140 B.t.u./hr.

7. Lower section, right wall:

Temperature on inside surface, # 16	259° F.
Temperature on outside surface, air	75° F.
Inner area	2.77 sq. ft.
Outer area	3.69 “ “
Log mean area	3.21 “ “
Thickness	4.50 in.
Mean brick temperature	167° F.
Conductivity at this temperature	0.76

Loss = 100 B.t.u./hr.

8. Lower section, front wall, except doorway:

Temperature on inside surface (15, 16, 19)	316° F.
Temperature on outside surface	100° F.
Inner area	1.70 sq. ft.
Outer area	2.83 “ “
Log mean area	2.22 “ “
Thickness	4.50 in.
Mean brick temperature	208° F.
Conductivity at this temperature	0.776

Loss = 83 B.t.u./hr,

9. Heat losses through doorway:

Temperature on inside surface, #17	650° F.
Temperature on outside surface, by thermocouple	150° F.
Inner area	1.44 sq. ft.
Outer area	1.44 " "
Log mean area	1.44 " "
Thickness	4.50 in.
Mean brick temperature	400° F.
Conductivity at this temperature	0.87

Loss = 139 B.t.u./hr.

10. Heat losses from bottom of furnace:

Average surface temperature	250° F.
Air temperature below furnace	90° F.
Heat loss (from Johns-Manville chart, Fig. 6)	2.43 B.t.u./hr./sq. ft./°F.
Area	7.03 sq. ft.

Loss = 2730 B.t.u./hr.

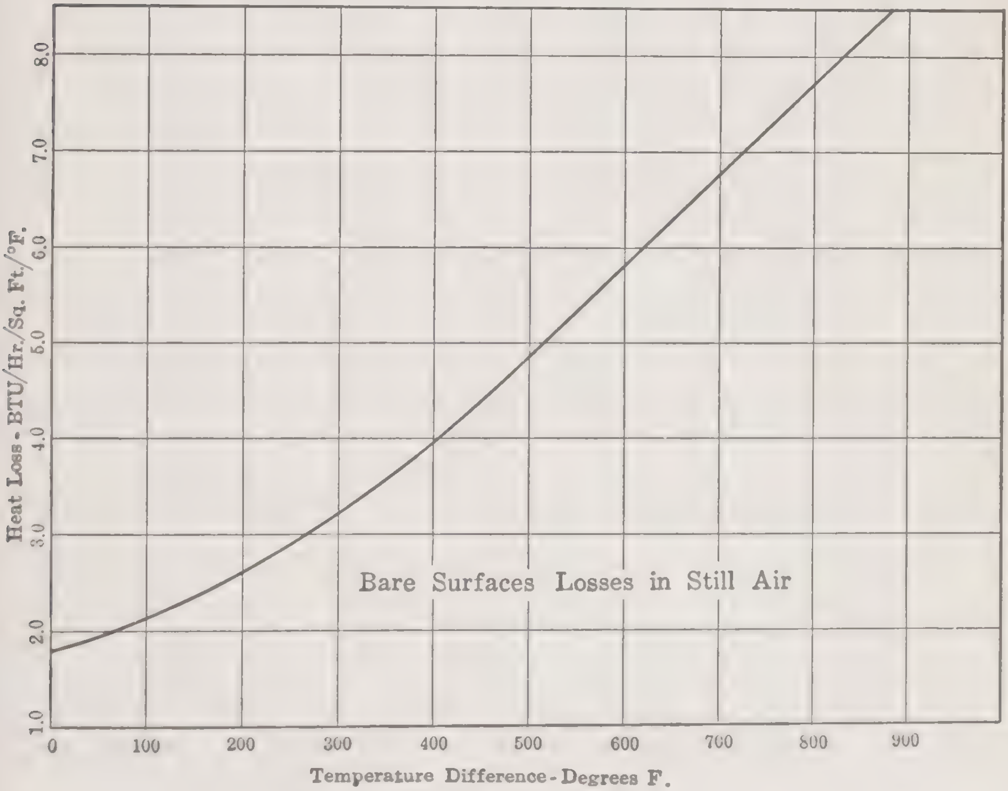

Fig. 6

11. Losses from top surface of furnace:

Average surface temperature	200° F.
Air temperature	70° F.
Heat loss (from Johns-Manville chart, Fig. 6)	2.28 B.t.u./hr./sq. ft./°F.
Area	6.21 sq. ft.

Loss = 1840 B.t.u./hr.

12. Losses from chimney surface:

Average surface temperature	350° F.
Air temperature	70° F.
Heat loss (from Johns-Manville chart, Fig. 6)	3.13 B.t.u./hr./sq. ft./° F.
Area	2.42 sq. ft.

Loss = 2120 B.t.u./hr.

Heat losses from furnace accounted for:

1. Upper section, front wall	302 B.t.u.
2. " " rear wall	308 "
3. " " left wall	401 "
4. " " right wall	369 "
5. Lower section, back wall	105 "
6. " " left wall	140 "
7. " " right wall	100 "
8. " " front wall	83 "
9. Doorway	139 "
10. Bottom of furnace	2730 "
11. Top surface	1840 "
12. Chimney surface	2120 "
Total accounted for losses	8637 B.t.u./hr.

Other losses, such as air infiltration, etc., cannot be accurately accounted for; so are listed as such in the final heat balance.

CALCULATION OF EFFECTIVENESS OF OUTER INSULATION LAYERS

Upper Section

	Losses through Wall	Couples	Drop	Thickness	Mean Temperature	Mean Area
Front	302	(23–27)	72° F.	2 in.	120° F.	5.59 sq. ft.
Rear	308	(22–26)	57° "	2 "	104° "	5.59 " "
Left	401	(24–28)	62° "	2 "	108° "	5.88 " "
Right	369	(25–29)	51° "	2.5 "	110° "	5.90 " "

k = B.t.u./hr./sq. ft./° F./in. thickness

Front wall — Johns-Manville improved Asbestocel

$$302 = k \times 5.59 \times 72/2 \quad \text{or} \quad k = 1.50 \text{ B.t.u./hr./sq. ft./° F./in.}$$

Rear wall — Johns-Manville improved Asbestocel

$$308 = k \times 5.59 \times 57/2 \quad \text{or} \quad k = 1.93 \text{ B.t.u./hr./sq. ft./° F./in.}$$

Left wall — Johns-Manville 85% magnesia block

$$401 = k \times 5.88 \times 62/2 \quad \text{or} \quad k = 2.19 \text{ B.t.u./hr./sq. ft./° F./in.}$$

Right wall — Armstrong Nonpareil N-16 insulating brick

$$369 = k \times 5.90 \times 51/2.5 \quad \text{or} \quad k = 3.06 \text{ B.t.u./hr./sq. ft./° F./in.}$$

It must be understood that the values for k found above are not those of the material itself, but rather of the installation. The temperature drops determined are those from one surface of the material to the other, while it is the practice in many tables to give conductivities from a refractory surface (to which the insulation is attached) to the temperature of the air outside. This would give a lower value for the conductivity, although total heat loss would be unchanged.

COMBUSTION CALCULATIONS

	Fuel Gas Analysis	Flue Gas Analysis
CO_2	1.0	4.0
CO	2.4	....
CH_4	64.2	....
C_2H_4	1.8	....
H_2	22.8	....
O_2	1.4	13.5
N_2	6.4	82.5

B.t.u. per cubic foot burned, 756, determined experimentally.
Gas enters at 70° F. and is burned with air at 70° F.
Amount of gas burned, 99 cu. ft. per hour.

Weight balance of combustion process—
Basis: 100 lb.-mols of dry fuel gas

1. Total gas burned:

CO_2	1.0×44	44 lb.
CO	2.4×28	67 "
CH_4	64.2×16	1027 "
C_2H_4	1.8×28	50 "
H_2	22.8×2	46 "
O_2	1.4×32	45 "
N_2	6.4×28	179 "
100-lb.-mols		1458 lb.

2. Weight of dry flue gas:

Carbon balance—

Output—Basis: 100 lb.-mols of dry flue gas

C in CO_2	4.0 lb.-atoms

Input—Basis: 100 lb.-mols of dry fuel gas

C in CO_2	1.0 lb.-atoms
C in CO	2.4 " "
C in CH_4	64.2 " "
C in C_2H_4	3.6 " "
Total C	71.2 lb.-atoms

Mols of dry flue gas formed $71.2/4.0 \times 100$ or 1778 lb.-mols.

Total dry flue gas:

$CO_2 = 0.040 \times 1778 =$ 71.2 lb.-mol or 3,133 lb.
$O_2 = 0.135 \times 1778 =$ 250.0 " " or 7,680 "
$N_2 = 0.825 \times 1778 =$ 1465 " " or 41,020 "
Total 1778 lb.-mols or 51,833 lb.

3. Weight of air introduced for combustion:

Nitrogen balance. Basis: 100 lb.-mols of dry fuel gas

N_2 in flue gas $= 0.825 \times 1778 = 1465$ lb.-mol
N_2 in fuel gas $= 0.064 \times 100 = 6.4$ " "
N_2 from air $= 1459$ lb.-mol

Dry air introduced $= 1459/0.79 = 1845$ lb.-mol, or $29 \times 1845 =$ 53,600 lb.

Molal humidity of air $= 0.004$
Water introduced $= 0.004 \times 1845 = 7.4$ lb.-mol or $18 \times 7.4 = 134$ lb.
Wet air introduced $= 1845 + 7.4 = 1852.4$ lb.-mol

4. Water vapor in flue gases:

Hydrogen balance. Basis: 100 lb.-mols dry fuel gas

Input:

H_2 in CH_4, 64.2×2	128.4 lb.-mol
H_2 in H_2	22.8 " "
H_2 in C_2H_4, 1.8×2	3.6 " "
H_2 in water vapor in air	7.4 " "
Total input	162.2 lb.-mol

Output:

In dry flue gas	0.0 lb.-mol
Water vapor in flue gas	162.2 " "
Total output	162.2 lb.-mol
	or 2920 lb.

Total wet flue gases = 1852.4 + 162.2 = 1947.6 lb.-mol
or 51,833 + 2920 = 54,753 lb.

5. Percentage excess air used:

Basis: 100 lb.-mols of dry fuel gas

Combustible Component		Oxygen Required
CO	2.4 lb.-mol	1.2 lb.-mol
CH_4	64.2 " "	128.4 " "
C_2H_4	1.8 " "	5.4 " "
H_2	22.8 " "	11.4 " "

Total oxygen required	146.4 lb.-mol
Present in fuel gas	1.4 " "
Oxygen from air	145.0 lb.-mol
Theoretical air 145/0.21 =	690 lb.-mol
Air supplied	1845 " "

Percentage excess = (1845 − 690)/690 × 100 = 167%

6. Calculation of lb.-mols of gas used from heating value:

Experimental heating value		756 B.t.u./cu. ft.
Heating value of:	CO	121,716 B.t.u./lb.-mol
	CH_4	383,022 " " "
	C_2H_4	597,600 " " "
	H_2	122,958 " " "

Heating value of 100 lb.-mols of dry flue gas:

CO	121,716 × 2.4	292,118 B.t.u.
CH_4	383,022 × 64.2	24,590,012 "
C_2H_4	597,600 × 1.8	1,075,680 "
H_2	122,958 × 22.8	2,803,442 "
Total per 100 lb.-mols		28,761,252 B.t.u.

$$99 \text{ cu. ft. } \frac{756 \times 99 \times 100}{28.761,252} \quad \text{or} \quad 0.260 \text{ lb.-mol}$$

7. Summary of Weight Balance of Combustion Process:

Basis: 99 cu. ft. of fuel gas/hour or 0.260 lb.-mol

Input:	Dry fuel gas	3.79 lb.
	Dry air	139.20 "
	Water vapor in air	0.35 "
	Total input	143.34 lb.
Output:	Dry flue gas	135.00 lb.
	Water vapor in flue gas	7.60 "
	Unaccounted for	0.74 "
	Total output	143.34 lb.

HEAT BALANCE OF ENTIRE FURNACE

Reference temperature 70° F. Room temperature?
Basis: 1 hour's run using 99 cu. ft. of fuel gas

Heat Input

1. Heating value of dry fuel gas
   $756 \times 99 = 74{,}840$ B.t.u.
2. Sensible heat of fuel gas
   Enters at base temperature
   0 B.t.u.
3. Sensible heat of dry air
   Enters at base temperature
   0 B.t.u.
4. Sensible heat of water vapor in air
   Dew point = 30.5° F.
   Molal heat of vaporization at 30.5 = 19,330 B.t.u./lb.-mol
   Heat content of water vapor is—
   $0.0194 \quad 19{,}330 - (18 \times 39.5) \quad (39.5 \times 8.3)$ or 367 B.t.u.
5. Heat content of water in pipe coil
   Inlet temperature = 47° F.
   Heat content $= 1 \times 372\,(47 - 70) = -8{,}556$ B.t.u.
6. Heat content of air in pipe coil

Entering temperature	74.5° F.
Weight of air	19.4 lb.
Molal humidity	0.004

Heat content is:

$$(74.5 - 70)(19.3)(0.24) \quad 19{,}330 - (18 \times 44) \quad 44 \times 8.3$$
$$0.00267 = 71 \text{ B.t.u.}$$

Heat Output

1. Heating value of flue gas:

No CO 0 B.t.u.

2. Heat content of dry flue gas:

Basis: 0.260 lb.-mol of fuel gas

$CO_2 = 0.040 \times 0.260 \times 1778/100 = 0.185$ lb.-mol
$O_2 = 0.135 \times 0.260 \times 1778/100 = 0.624$ “
$N_2 = 0.825 \times 0.260 \times 1778/100 = 3.816$ “

Heat contents:

$CO_2(670 - 70)(0.185)(9.90) = 1{,}100$ B.t.u.
O_2, $N_2(670 - 70)(4.44)(7.10) = 18{,}930$ “

Total heat............... 20,030 B.t.u.

3. Heat content of water vapor in flue gases:
Molal humidity 162.2/1778 = 0.0912
Dew point 108° F.
Heat of vaporization at 108° F., 18,560 B.t.u./lb.-mol
Mean molal heat capacity 8.56
Heat content:

$$0.422 \quad 18{,}560 \quad (38 \times 18) \quad (562 \times 8.56) = 10{,}150 \text{ B.t.u.}$$

4. Heat output in piped water:

$372(113 - 70) = 15.996$ B.t.u.

5. Heat content of piped air:

$$(540 - 70)\ (7.06)\ (19.3/29) \quad 19{,}330 - (18 \times 44)$$
$$(510.5 \times 8.5) \quad 0.00267 = 2271 \text{ B.t.u.}$$

Summary of Heat Balance

Input

1. Heating value of dry fuel gas.......................	74,840 B.t.u.
2. Sensible heat of dry fuel gas.......................	0 “
3. “ “ “ “ air...........................	0 “
4. Heat content of water vapor in air.................	367 “
5. “ “ “ piped water......................	−8,556 “
6. “ “ “ “ air.........................	71 “
Total input..............................	66,722 B.t.u.

Output

1. Heating value of flue gas	0	B.t.u.
2. Heat content of dry flue gas	20,030	“
3. “ “ “ water in flue gas	10,150	“
4. “ “ “ piped water	15,996	“
5. “ “ “ “ air	2,271	“
6. “ losses from walls accounted for	8,637	“
7. Unaccounted for losses	9,638	“
Total output	66,722	B.t.u.

EXPERIMENT NO. 18

QUALITY OF STEAM; BY THE THROTTLING CALORIMETER

Experiments in which steam is used as the heating medium require a determination of the quality of steam, as the heating value per pound of the steam varies as its quality varies.

Object. To determine the quality of a steam sample.

The percent by weight of steam in a mixture of steam and water is the quality of the steam. The apparatus used to determine the moisture content of steam is called a calorimeter, a misuse of the term, as the name is not descriptive of the function of the apparatus. Modern steam calorimeters are, in general, of the throttling or separatory type.

The instrument selected for this particular experiment is a simple compact calorimeter of the throttling type.

"This calorimeter consists of two concentric metal cylinders [1] screwed to a cap containing a thermometer well. The steam pressure is measured by a gauge placed in the supply pipe or the orifice A and expands to atmospheric pressure, its temperature at this pressure being measured by a thermometer placed in the cup C. To prevent as far as possible radiation losses, the annular space between the two cylinders is used as a jacket, steam being supplied to this space through the hole B."

When steam passes through an orifice from a higher to a lower pressure, as is the case with the throttling calorimeter, no external work has to be done in overcoming a resistance. Hence, if there is no loss from radiation, the quantity of heat in the steam will be exactly the same after passing the orifice as before passing. If the higher steam pressure is 160 pounds gauge and the lower pressure that of the atmosphere, the total heat in a pound of dry steam at the former pressure is 1195.9 B.t.u. and at the latter pressure 1150.4 B.t.u., a difference of 45.4

---

[1] This description of the steam calorimeter and its use has been taken from "Steam," The Babcock & Wilcox Company.

B.t.u. As this heat will still exist in the steam at the lower pressure, since there is no external work done, its effect must be to superheat the steam. Assuming the specific heat of superheated steam to be 0.47, each pound passing through will be superheated $\frac{45.4}{0.47} = 96.6$ degrees. If, however, the steam had contained one percent of moisture, it would have contained less heat units per pound than if it were dry. Since the latent heat of steam at 160 pounds gauge pressure is 852.8 B.t.u., it follows that the one percent of moisture would have required 8.5 B.t.u. to evaporate it, leaving only $45.4 - 8.5 = 36.9$ B.t.u. available for superheating; hence, the superheat would be $\frac{36.9}{0.47} = 78.5$ degrees, as against 96.6 degrees

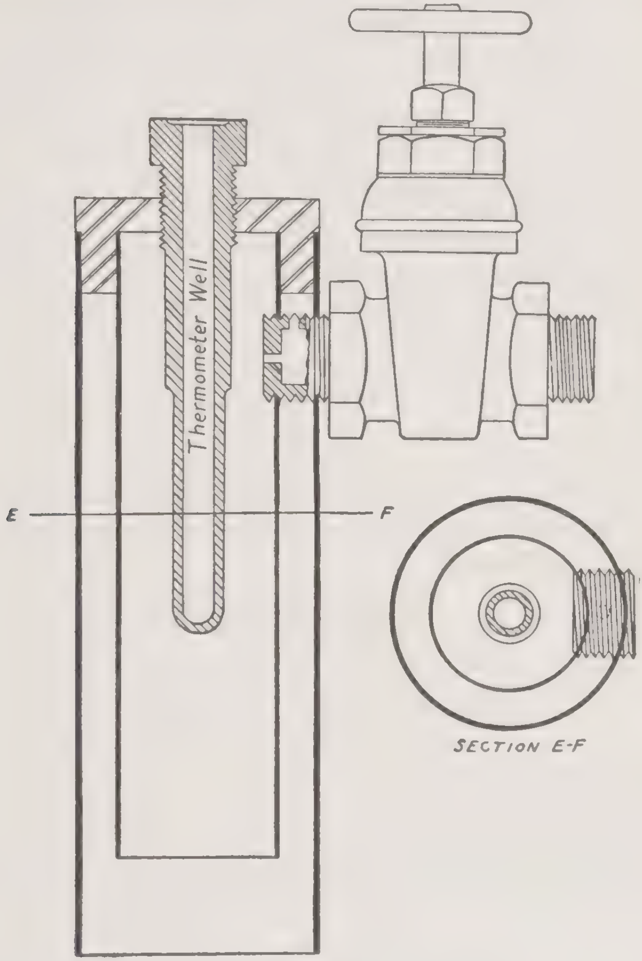

FIG. 7

for dry steam. In a similar manner, the degree of superheat for other percentages of moisture may be determined. The action of the throttling calorimeter is based upon the foregoing facts, as shown below.

Let H = total heat of 1 lb. of steam at boiler pressure
L = latent heat of steam at boiler pressure
h = total heat of steam at reduced pressure after passing orifice
t_1 = temperature of saturated steam at the reduced pressure
t_2 = temperature of steam after expanding through the orifice in the disc
0.47 = specific heat of saturated steam at atmospheric pressure
x = proportion by weight of moisture in steam

The difference in B.t.u. in a pound of steam at the boiler pressure and after passing the orifice is the heat available for evaporating the moisture content and superheating the steam. Therefore,

$$H - h = xL + 0.47(t_2 - t_1)$$

or

$$x = \frac{H - h - 0.47(t_2 - t_1)}{L}$$

Almost invariably the lower pressure is taken as that of the atmosphere. Under such conditions, $h = 1150.4$ and $t_1 = 212$ degrees. The formula thus becomes:

$$x = \frac{H - 1150.4 - 0.47(t_2 - 212)}{L}$$

SAMPLE DATA AND CALCULATIONS

Temperature in throttle	214° F.
Steam pressure	100 lbs.
Temperature of steam	337.9° F.
Heat content of steam (from tables)	1189.3 B.t.u. per lb.
Latent heat of steam (from tables)	880.8 " " "

x = moisture content, in percent.

then:

$$x = \frac{1189.3 - 1150.4 - .47\,(337.9 - 214)}{880.8} \times 100$$

$$x = \frac{19.3}{880.8} \times 100 = 2.2\% \text{ moisture in steam.}$$

The steam is therefore 97.8% *steam.*

EXPERIMENT NO. 19

QUALITY OF STEAM; BY THE BARREL CALORIMETER

Object. To determine the quality of a steam sample.

Procedure. The barrel calorimeter method of determining the quality of steam is as follows: A metal or wooden barrel is filled with a weighed amount of water, two to three hundred pounds, and the temperature is taken. Steam is now passed through the water for a definite length of time. The temperature is again taken and the water again weighed. The increase in weight divided by the time gives the pounds of steam per unit length of time. The increase in temperature multiplied by the total pounds of water will give the number of B.t.u. in the steam. This number divided by the increase in weight will give the B.t.u. per pound of steam.

Knowing the pressure of the steam, the B.t.u. content per pound of steam can be found in the steam tables. Divide the difference in heat contents by the larger and multiply by one hundred. Subtract this number from one hundred and this is the percentage of dry steam.

$$\text{B.t.u. in steam} = (t_2 - t_1)W$$

$$\text{B.t.u. per lb. steam} = W(t_2 - t_1)/(W_2 - W_1)$$

$$\%\ \text{dry steam} = 100 - \frac{(\text{Theoretical B.t.u.} - \text{Exp. B.t.u.} \times 100)}{\text{Theoretical B.t.u.}}$$

SAMPLE DATA AND CALCULATIONS

Steam pressure 92 lbs. = 106.7 lbs. absolute
Initial temperature of water = 23° C.
Final " " " = 85° C.
Initial weight of water = 202 lbs.
Final " " " = 228 "
Weight steam condensed = 26 "
Temperature of steam = 332.8° F.
Latent heat of steam = 882.1 B.t.u. per lb.

Calculations:

$$\text{Heat in steam theoretical}\ldots\left\{\begin{array}{r} 26 \times 882.1 = 22{,}950 \quad \text{B.t.u.} \\ 26(212 - 185) = \phantom{22{,}}702.5 \qquad\quad \\ \hline 23{,}652.5 \qquad\quad \end{array}\right.$$

Actual B.t.u. obtained.........$202 \times 111.5 = 22{,}550$

Percent dry steam:

$$-\frac{(23{,}652.5 - 22{,}550) \times 100}{23{,}652.5} + 100 = 95.33\%\ \text{dry steam}$$

EXPERIMENT NO. 20

Condensing Vapor to Liquid Flowing in an Inner Pipe

REFERENCES

Morris and Whitman, Industrial and Engineering Chemistry, Vol. 20.
Dittus and Boelter, University of California publications in engineering, Vol. 2, p. 443.
Sherwood and Petrie, Industrial and Engineering Chemistry, Vol. 24, p. 736.

The flow of heat from a condensing vapor to a liquid flowing on the other side of a retaining wall is one of the most important applications of heat transmission. Calculations involved in the design of condensers, coolers, heat exchangers and evaporators are based on the formulae developed by investigators of this type of flow of heat.

The available knowledge on the subject appears to be very meager when we consider the fact that with years of investigation, the only formula regarded as dependable in calculating the film coefficient for condensing vapors applies only to horizontal pipes. If the surface be vertical or lying at an angle, the calculations are subject to considerable question as to accuracy.

Much additional experimental investigation is needed to establish the accuracy and application of various formulae proposed for these calculations but which, to date, have not been sufficiently tested against experimental data.

Investigators, until the last few years, have assumed that film condensation was the normal and customary form in which most vapors condense on most cooling surfaces. Recent investigations have cast considerable doubt on this assumption, as many vapors have been found condensing on many surfaces as drops rather than as a film.

Conditions inducing one type of condensation, in exclusion of the other, are under study, but not very well understood as yet. The careful investigator does not assume, therefore, that film condensation is occurring, but arranges the equipment so that some sections, at least, of the condensing area are visible and thus knows the kind of condensation occurring.

The conditions selected for this experiment are those for which our formulas are accepted as accurate and much experimental work in our own laboratories has demonstrated that the careful experimenter can correlate theoretical calculations and experimental results.

Object. Determination of the overall coefficient of heat transfer, any vapor to any liquid, through an intervening metal wall, and of the respective resistivities to heat flow of the various parts of the transfer system, in so far as this is possible. Verification of the various film coefficients for heat transfer, as published in the literature.

Equipment. The equipment used in this experiment consists essentially of the following parts: An inner tube through which cooling liquid is flowing, the cooling liquid passing first through a fluid meter, then through the inner tube which, at the outflow end, is baffled and provided with a thermocouple for measuring the temperature. The outflowing liquid is then delivered to a tank where it can be weighed to check the readings of the fluid meter.

The outer tube is the one through which the vapor flows, and, in the same direction as the liquid. The vapor line passes through a trap where condensate is removed, then through a cast ring extending around the tube and acting as a vapor distributor, and into the exterior jacket of the condenser tube. Just prior to the entrance of the vapor into the condenser tube jacket are located a pressure gauge and a thermocouple.

Both tubes slope very slightly toward the discharge end; and at the discharge end of the inner tube is located a thermocouple for determining outflowing liquid temperature. The condensate from the jacket flows into a small condensate receiver then out through a Sarco steam trap.

Procedure. The condensate temperature is taken, as discharged, and the amount of condensate is determined by weighing it.

Four thermocouples are installed at equal intervals along the inner pipe through which the liquid is flowing. These thermocouples are used in measuring the temperature of the tube wall, as they are placed midway between the exterior and interior surfaces of the tube wall.

The couples must be selected of these metals not likely to rust: Copper-constantan, chromel-alumel, and nickel-nickel chromium have been found satisfactory. The lead wires are Bakelite coated to minimize heat transfer to or from the leads. The construction is shown in detail on the accompanying cut, Fig. 8.

PRELIMINARY REPORT

It is essential that, prior to attempting this experiment, the student become informed as to operating conditions required for securing data upon which accurate calculations may be based.

There is considerable material in the literature of recent times, detailing the experiments conducted by various investigators seeking to verify the constants to be calculated.

The authors cited in the references at the opening of this experiment deal primarily with the computations connected with the determination of the liquid film coefficient.

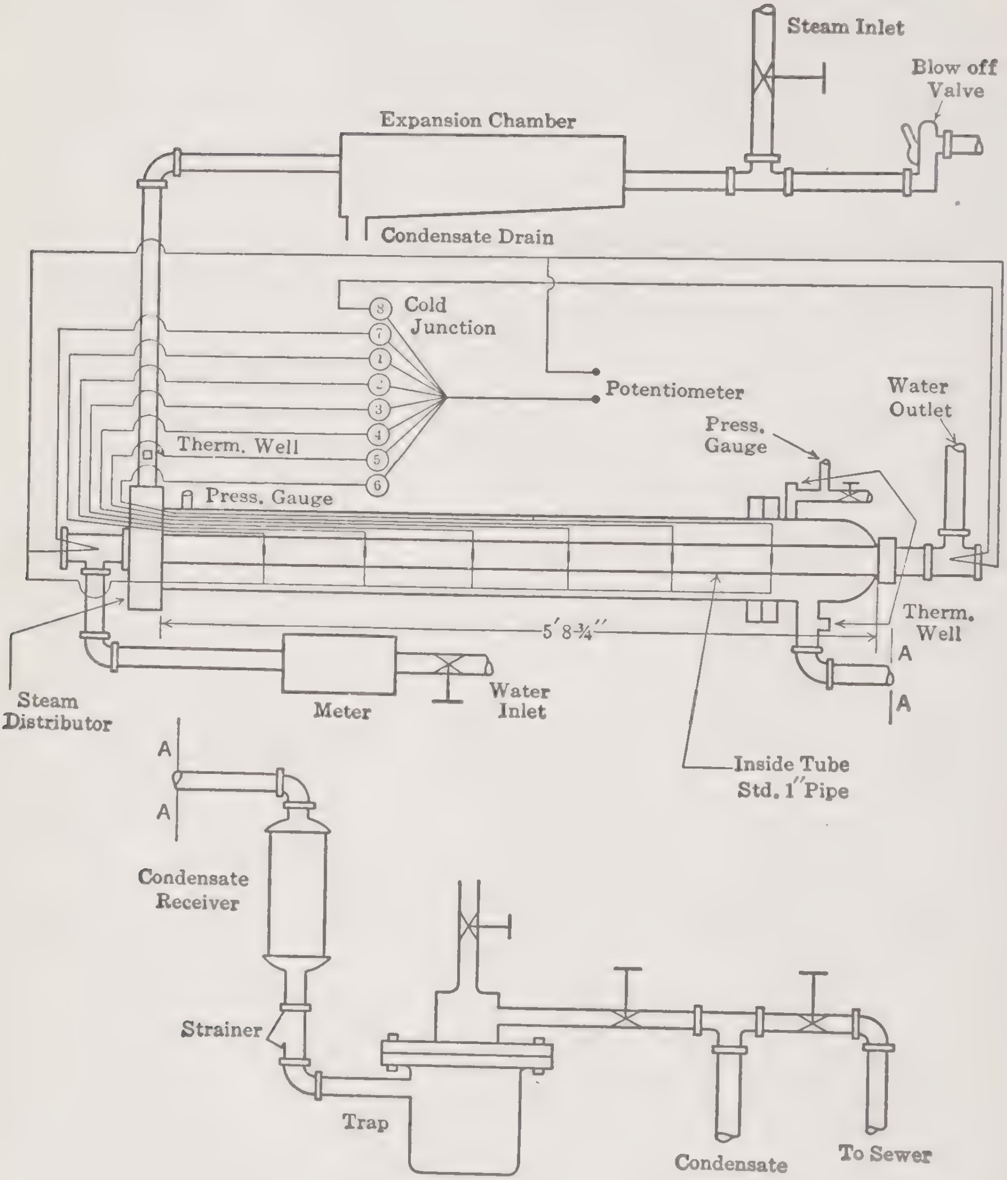

FIG. 8.—Heat transfer from condensing vapor.

The investigations as to the calculations of the film coefficient for condensing vapor have been largely done abroad, the leading investigator being Nusselt. The past ten years have been noted by the publication of many such investigations made in the laboratories of this country and the most informative of the recent work is to be found in American publications. Considerable comment on Nusselt's methods of calculation is to be found in the current literature. Some of the articles are: Monrad and Badger, Industrial and Engineering Chemistry, Vol. 22, page 1103; Colburn and Haugen, University of Wisconsin, Engineering Experiment Station Bulletin 70.

It is noted that Reynolds' criterion is utilized in each of the formulas referred to. Consult Badger and McCabe as to a definition of Reynolds' criterion and its derivation, also reread the introduction to the experiments on flow of heat.

Explain how it is expected to make use of these theoretical equations in solving the problems of quantity of heat transfer involved in the present problem, state the theoretical equations applicable to the particular operating conditions.

Outline, briefly but concisely, the methods to be used in carrying out the test. Use as a basis for this outline, the data sheets supplied with these instructions and the information which has been gathered from consultation of the references cited. Using no figures, indicate the methods of calculation which will be attempted. It is to be noted that no preliminary report will be accepted on this specific experiment, unless such report shows clearly a definite comprehension of the essential data required for making the calculations desired.

REPORT

Supply the information indicated in the sample data sheet accompanying the preliminary report. Show, in complete detail, all the calculations made from these data.

Discuss the experiment from the standpoint of data which might be in error, possible effects of these errors on the calculations and state any conclusions reached as to causes for divergence of the calculated theoretical coefficients from those determined experimentally.

State, for each formula used, an opinion as to its reliability, such opinions being based on the references consulted.

Assume that 500 pounds of steam at 250 pounds gauge pressure are to be condensed per hour and cooled to 160° F. Calculate, from your data, the required condenser area. Velocities and area are not to be changed.

How much cooling water, entering at 55° F. and leaving at 140° F., will be required? Velocity the same as in the experimental data.

SAMPLE DATA AND CALCULATIONS
(Milleville-Robbie)

The sample data and calculations are given for two consecutive experiments in which one was film condensation, the other drop wise.

	Film	Drop Wise
Duration of test, hrs	1.0	1.0
Steam gauge pressure inlet, lbs	12.7	12.7
Inlet steam temp. (thermometer)	244.0	255.0
“ “ “ (from pressure)	245.2	245.2
Degrees superheat	......	9.8
Steam gauge pressure, outlet	12.1	12.1
Outlet steam temp. (thermometer)	242.8	243.0
“ “ “ (from pressure)	243.9	243.9
Weight of condensate, lbs	69.5	69.0
Weight of water, lbs	1585.0	1578.0
Initial temperature of water	55.4	56.0
Final “ “ “	96.8	97.5
Rise in temperature	41.4	41.5
Pipe temperature at Couple No. 1	226.0	231.3
“ “ “ “ “ 2	221.0	227.7
“ “ “ “ “ 3	221.5	226.8
“ “ “ “ “ 4	216.0	221.9
“ “ “ “ “ 5	221.9	228.0
“ “ “ “ “ 6	218.3	209.3
Material of condenser tube	Monel metal	

Tube dimensions (same for both):

Length, 5 ft 8.75 in. = 5.73 ft.
Inner diameter, 1.049 in. = 0.0874 ft.
Outer diameter, 1.315 in. = 0.1096 ft.
Heat transfer area (arith. mean) = 1.77 sq. ft.

Film Condensation:

Experimental Coefficients:

$$U = \frac{Q}{A\,\Delta t}, \qquad Q = 65{,}600 \text{ B.t.u.}, \quad A = 1.77 \text{ sq. ft.}$$

$$\Delta t = \frac{(244 - 55.4) - (242.8 - 96.8)}{2.303 \log \dfrac{188.6}{146.0}} = \frac{42.6}{2.303 \times 0.1115} = 166$$

$$U = \frac{65{,}600}{1.77 \times 166} = 223 \text{ B.t.u.}$$

$$hs = \frac{Q}{A\,\Delta t}, \qquad Q = 65{,}600$$

$$A = 0.1096 \times \pi \times 5.73 = 1.974 \text{ sq. ft.}$$

There is no direct information with respect to Δt. It therefore becomes necessary to compute it.

The average temperature at the mid point of the pipe wall is known; 221°.

The same quantity of heat is flowing from the outer pipe surface to the mid point as is flowing from the mid point to the inner surface: Let Δt_1 = temperature difference between mid point and outer surface and Δt_2 = temperature difference between mid point and inner surface. L = thickness in ft. of pipe wall from mid point to either surface = 0.00555 ft.

$$\Delta t_1 = \frac{Q \times L}{kA_1} \qquad \Delta t_2 = \frac{Q \times L}{kA_2}$$

$Q = 65{,}600,\quad L = 0.00555,\quad k = 16.7$

$A_1 = 1.90$ sq. ft. $\quad A_2 = 1.70$ sq. ft.

$$\Delta t_1 = \frac{65{,}600 \times 0.00555}{16.7 \times 1.90} = 11.5°$$

$$\Delta t_2 = \frac{65{,}600 \times 0.00555}{16.7 \times 1.70} = 12.8°$$

Outer pipe wall temperature, $221 + 11.5 = 232.5°$
Inner pipe wall temperature, $221 - 12.8 = 208.2°$
Δt (steam to inner pipe wall) $= 244 - 232.5 = 11.5°$

$$hs = \frac{65{,}600}{1.97 \times 11.5} = 2880$$

Water Film Coefficient:

$$hw = \frac{Q}{A\Delta t} \qquad Q = 65{,}600, \qquad A = 1.57 \text{ sq. ft.}$$

$$\Delta t = \frac{(208.2 - 55.4) - (208.2 - 96.8)}{2.303 \log \dfrac{152.8}{111.4}} = \frac{41.4}{2.303 \times 0.137} = 131°$$

$$hw = \frac{65{,}600}{1.57 \times 131} = 319 \text{ B.t.u.}$$

Theoretical Coefficient:

Steam Film Coefficient:

$$hs = 0.725\left(\frac{k^3\rho^2 gr}{D\mu\Delta t}\right)^{\frac{1}{4}}$$

Average temperature of Vapor and Pipe Surface is 232° F.

$k = 0.42$ $\qquad k^3 = 0.0741$

$\rho = 59.4$ $\qquad \rho^2 = 3525$

$g = 4.18 \times 10^8$

$r = 949.7$ (at 244° F.)

$D = 0.1097$

$\mu = 2.42 \times 0.20 = 0.484$

$\Delta t = 11.5$

$$hs = 0.725 \left[\frac{0.0741 \times 3525 \times 4.18 \times 10^8 \times 949.7}{0.1096 \times 0.484 \times 11.5}\right]^{1/4}$$

$$= 0.725[1.695 \times 10^{14}]^{1/4}$$

$$= 0.725 \times 3605$$

$$= 2615$$

Water Film Coefficient:

$$hw = \frac{k}{D}\, 0.0225 \left(\frac{DG}{\mu}\right)^{0.8} \times \left(\frac{Cp\mu}{k}\right)^{0.4}$$

Average temperature of water = 76.1° F.

$k = 0.344$

$D = 0.0874$

$\mu = 2.42 \times 0.93 = 2.25$

$Cp = 1.00$

$$G = \frac{1585}{\pi \times \dfrac{0.0885^2}{4}} = 257{,}900$$

$$hw = \left(\frac{0.344 \times 0.0225}{0.0874}\right) \times \left(\frac{0.0874 \times 257{,}900}{2.25}\right)^{0.8} \times \left(\frac{1 \times 2.25}{0.344}\right)^{0.4}$$

$$= (0.0885) \times (10{,}000)^{0.8} \times (6.54)^{0.4}$$

$$= 0.0885 \times 1580 \times 2.12$$

$$= 296$$

Overall Coefficient:

$$U = \frac{1}{\frac{1}{hs} + \frac{L}{k} + \frac{1}{hw}}$$

$hs = 2625$ $k = 16.7$

$L = 0.011$ $hw = 296$

$$U = \frac{1}{\frac{1}{2625} + \frac{0.011}{16.7} + \frac{1}{296}} = \frac{1}{0.000381 + 0.00068 + 0.003880}$$

$$= 225$$

Dropwise Condensation:

Experimental Coefficients:

The Overall Coefficient:

$$U = \frac{q}{A\Delta t}$$

$q = 65{,}400$

$A = 1.77$

$$\Delta t = \frac{(245.2 - 56.0) - (243.9 - 97.5)}{2.303 \log \frac{189.2}{146.4}} = \frac{42.8}{0.256} = 167$$

$$U = \frac{65{,}400}{1.77 \times 167} = 229$$

Steam " Film " Coefficient:

$$hs = \frac{q}{A\Delta t}$$

$q = 65{,}400$

$A = 1.974$

$$\Delta t:\ \Delta t_1 = \frac{65{,}400 \times 0.00555}{16.7 \times 1.90} = 11.5$$

$$\Delta t_2 = 12.8$$

Outer pipe temperature = 224 + 11.5 = 235.5

$$\Delta t = 244.5 - 235.5 = 9.0 \qquad hs = \frac{65{,}400}{1.974 \times 9.0} = 3{,}680$$

Water Film Coefficient:

$$hw = \frac{q}{A\,\Delta t}$$

$q = 65{,}400$

$A = 1.57$

Δt: Inner pipe temperature = 224 − 12.8 = 211.2

$$\Delta t = \frac{(211.2 - 56.0) - (211.2 - 97.5)}{2.303 \log \dfrac{155.2}{113.7}} = \frac{41.5}{0.313} = 132.5$$

$$hw = \frac{65{,}400}{1.57 \times 132.5} = 314$$

Theoretical Coefficient:
Steam Film Coefficient:

$$hs = 0.725 \left[\frac{k^3\rho^2 gr}{D\mu\Delta t}\right]^{1/4}$$

Only change from that of filmwise is Δt from 11.5 to 9.0

$$hs = 0.725 \left[1.695 \times \frac{11.5}{9.0} \times 10^{14}\right]^{1/4} = 0.725 \times 3830 = 2780$$

Water Film Coefficient:

$$hw = \frac{k}{D}\, 0.0225 \left(\frac{DG}{\mu}\right)^{0.8} \left(\frac{Cp\mu}{k}\right)^{0.4}$$

same as before

$= 296$

Overall:

$$U = \frac{1}{\dfrac{1}{hs} + \dfrac{L}{k} + \dfrac{1}{hw}}$$

$$= \frac{1}{\dfrac{1}{2780} + \dfrac{0.00111}{16.7} + \dfrac{1}{296}} = \frac{1}{0.00442}$$

$= 226$

EXPERIMENT NO. 21

Flow of Heat from Condensing Benzene Vapors, Including a Study of Pipe-Wall Temperature Around the Tube

Object. To study temperature variance in a horizontal condenser tube. It has come to be accepted as a fact that, with a horizontal condenser tube, the tube wall temperature varies around the perimeter. It is thought advisable, therefore, to present a type of equipment and a method of operation with which such items may be studied.

Equipment. The condensing tube is of highly polished Monel metal, outside diameter 1.312 inches, inner diameter 1.049 inches, length exposed to vapors 67.0 inches.

The outer jacket is a stock size piece of Pyrex piping 3.5 inches in diameter and 60.0 inches long. The pipe has bell ends, held in a clamp and gasket, and this in turn held by stud bolts, against the flat, vertical face of the casting, forming the vapor inlet ring and the condensate outlet.

The outside face of each of these end castings is tapped, fitted with a packing gland on the water inlet end, and with a special flanged face plate on the casting at the water outlet end.

The thermocouple wires are brought out between two gaskets lying between the flange plates. The flange bolts are 45 degrees apart, so by taking out the bolts the tube can be rotated 45 degrees, changing the thermocouple locations by this amount, then bolted in place in the new position.

The connections at the ends of the water tube are made by inserting the water lines inside the Monel tube, then the joint made tight by the use of a packing nut.

A crumpled wire screen is inserted in the water pipe, near the outlet end, to assure thorough mixing at the point where the temperature of the outlet water is taken.

A pressure gauge and a petcock to vent non-condensible gases are tapped into the distributor casting on the outlet end.

All temperatures are taken with thermocouples calibrated to 0.1° C.

The thermocouples used to measure the pipe-wall temperature are installed by a method similar to that described by Hebbard and Badger.[1] The method used is illustrated in Fig. 9. A chord hole is drilled through the center of the pipe wall, with a drill 0.0465 inch in diameter (No. 56). A radial hole is then drilled so that it meets the chord hole in the middle. The thermocouple wires, of No. 22 B. & S. gauge, are inserted in the chord hole, one from each side. Solder is dropped through the radial hole to form the junction. The excess solder is removed and the exterior surface polished with fine French polishing sandpaper.

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[1] G. M. Hebbard and W. L. Badger, "Measurement of Tube-Wall Temperatures in Heat Transfer Experiments," *Ind. Eng. Chem., Anal. Ed.*, 5, 359 (1933).

Before installation, the thermocouples are insulated with several baked-on layers of a methanol solution of bakelite. This insulation keeps the wires from touching the pipe wall, thus preventing a short circuit, and also minimizing heat flow to the thermocouple junction.

The thermocouple leads are brought out at one end of the tube between the two flange gaskets. The tube, the outer flange with the two gaskets, and the thermocouples acted as a unit rotating simultaneously. There were six thermocouples installed in the pipe wall, spaced 11.5 inches apart.

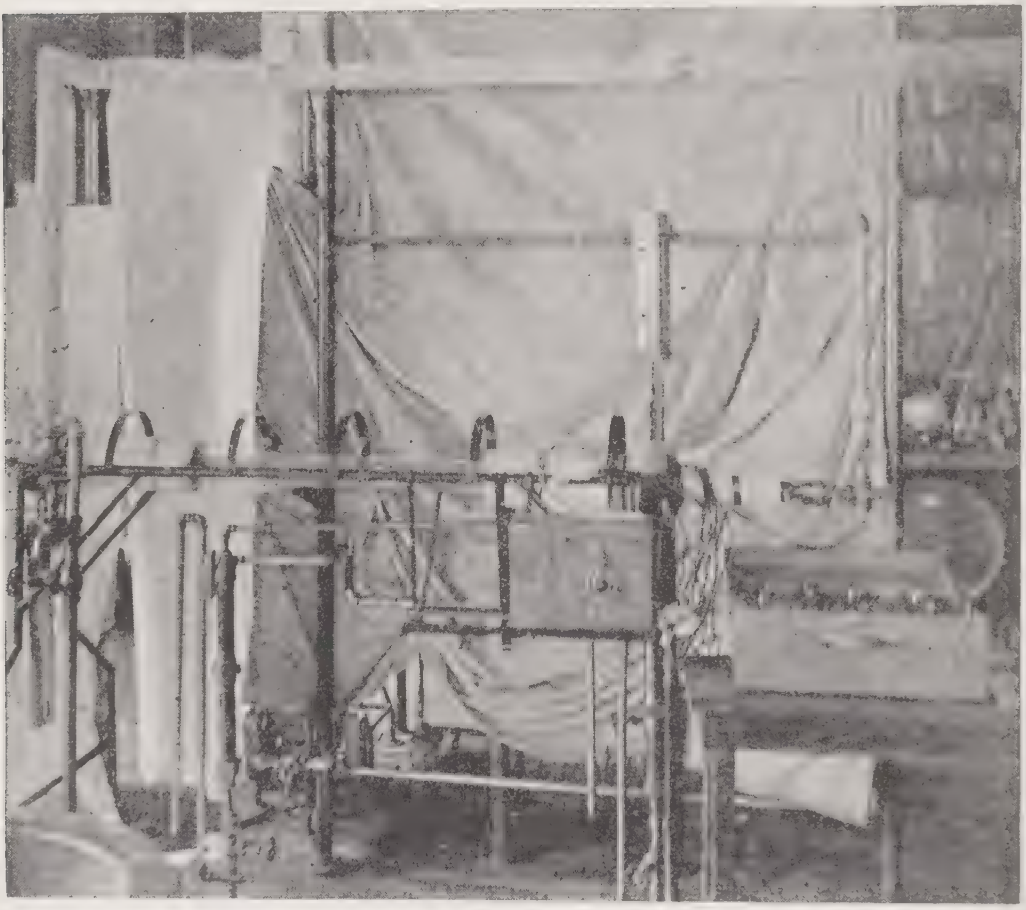

Fig. 9.—Heat transfer tube.

The assembly is shown in the photograph, Fig. 9, and the details in the line drawing, Fig. 10.

Procedure. About 40 gallons of technically pure benzene are placed in the steam heated still kettle. The steam is turned on, and at the same time the cooling water is started and allowed to flow at a rate of about 1000 pounds per hour. When the benzene vapors begin to condense on the tube, the type of condensation is carefully observed. When filmwise condensation is taking place along the entire length of the tube, and the system has attained equilibrium, the experiment is started.

The first set of data is taken with the tube rotated so that the thermocouples are located at the lowest point on the perimeter (the bottom of the tube). This point is designated in the data as zero degrees (0°), and all angles are measured from this point.

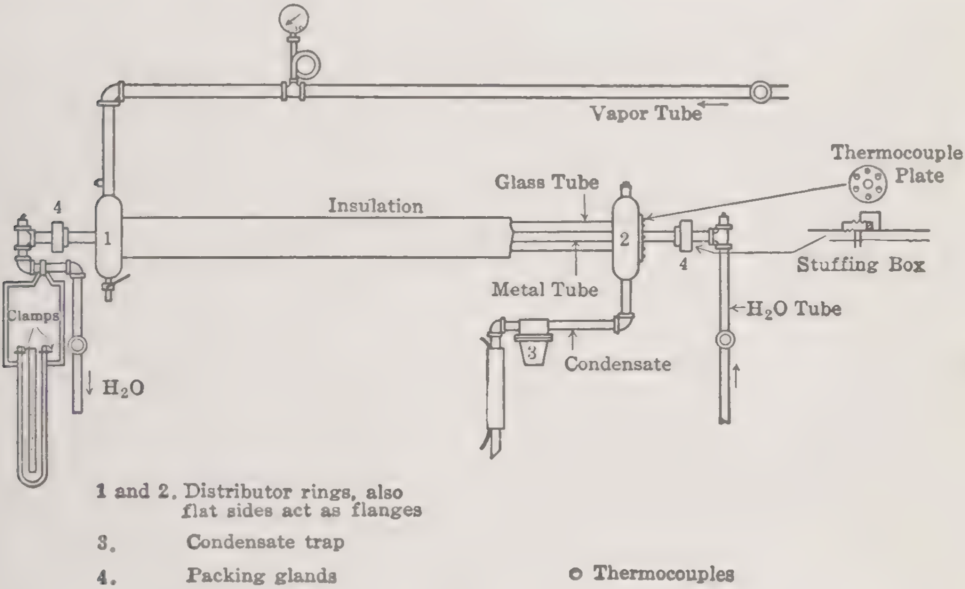

FIG. 10

The data taken are:

1. Temperature of the incoming water
2. Temperature of the outgoing water
3. Temperature of the vapor at both ends of the tube
4. Temperature of the tube wall at each of the thermocouple locations
5. Pressure of the vapor
6. Readings of the manometer for the orifice meter
7. Angle of rotation.

After the data are recorded, the tube is rotated through 45 degrees. After equilibrium has again been reached, the data are again taken. This is continued until one revolution of the tube has been completed.

A run is considered one complete revolution of the tube.

Thermocouple readings are taken with a Leeds & Northrup type K potentiometer. The temperatures were accurate to the nearest one-tenth of a degree C.

SAMPLE DATA AND CALCULATIONS

Fundamental data for seven runs with benzene as the condensing liquid are given in the following table.

TABLE 1

FUNDAMENTAL DATA FOR THE HORIZONTAL TUBE*

Run Number	2	3	4	5	6	7	8
Pounds of water per hour	1360	1155	1095	1060	1120	1080	1120
Temp. of water in	4.9	6.7	7.8	7.7	7.4	3.1	7.6
Temp. of water out	14.9	18.1	19.7	19.8	18.3	14.2	18.6
Pipe-surface temperature†	48.3	57.0	59.2	59.9	59.9	57.5	60.8
Vapor temp	80.2	80.2	81.6	81.6	82.4	79.8	82.4
Δt through film	31.9	24.0	22.4	21.7	22.5	22.3	21.6
Pressure, gage	atm.	½	1	1	1½	¼	1½
h (experimental)	222	288	303	294	282	280	297
h' (Nusselt)	286	306	312	314	211	311	314
h/h'	0.78	0.94	0.97	0.94	0.91	0.90	0.95

* All temperatures are given in degrees C.

† Calculated by the method discussed in Exp. 20, then averaged for the perimeter of the tube, as shown in Table 2.

TABLE 2

DATA FOR THE VARIATION OF PIPE-WALL TEMPERATURE*

Run Number							
0°†	41.1	48.0	45.8	48.2	48.8	47.0	51.6
45°	44.8	52.1	54.6	55.0	54.2	52.1	55.1
90°	48.4	56.4	58.9	59.1	59.6	56.8	60.9
135°	48.9	59.0	62.5	62.8	60.9	59.9	62.5
180°	49.2	60.9	64.5	65.3	64.6	62.2	63.6
225°	48.9	59.0	61.3	....	61.8	59.8	....
270°	48.5	56.4	57.8	....	57.5	56.3	....
315°	44.8	51.9	53.8	....	55.5	52.2	....
360°	40.8	47.0	49.5	....	51.6	47.4	....
Average angle	67°	81°	83°	81°	81°	81°	74°
Average temperature	46.7	55.5	57.7	58.4	58.4	56.0	59.3

* All temperatures given in degrees C.

† All angles are measured from the bottom of the perimeter.

The values obtained for the heat transfer coefficient of benzene range from 222 to 303 B.t.u. per hour per square foot per degree F. The value of 222 is in error because the operating conditions changed during the run. Disregarding this one value, the other values range from 280 to

303, the average being 290. It is evident from the data that the results do not cover a wide range of operating conditions.

The experimental values check rather well with those calculated by means of Nusselt's equation for the horizontal tube. An inspection of the data will show a variation of from three to eleven per cent between the two values, the experimental values being lower in every case. The same situation has been found to exist in the case of condensing steam. Thus, for a horizontal tube, Nusselt's equation can be depended upon to give satisfactory predictions for the value of the heat transfer coefficient, both for benzene and for steam.

The variables in Nusselt's equation were evaluated at the film temperature, which was taken to be the arithmetic average between the vapor temperature and the pipe-wall temperature. The equation is given below. The symbols in this equation are defined in the Flow of Heat Code of The American Institute of Chemical Engineers.

$$h = 0.725\left(\frac{k^3p^2gr}{uD\Delta t}\right)0.25$$

The curves plotted from the data given are symmetrical about the ordinate representing the top of the tube, indicating a corresponding symmetry in the pipe-wall temperature on both sides of the tube from the top to the bottom.

The pipe-wall temperature is a maximum at the top and a minimum at the bottom. The difference in temperature between these points ranges from 12.9° to 18.7° C.

There is a point at which a thermocouple can be located so that it can be depended upon to indicate the correct mean tube temperature. For these experiments the point lies at an angle of 99-100 degrees from the top of the tube. This angle was determined in the following manner: The area under the curve, found by graphical integration, was divided by the abscissa representing 360 degrees. This gave the value of the ordinate for the average tube-wall temperature. The intersection of this ordinate with the curve gave the value of the corresponding angle. It is very likely that this angle is specific to benzene vapors condensing on a tube of this diameter. However, it is just as likely that the angle for any vapor condensing on a tube of any diameter will not vary from this angle by more than a few degrees.

The shape of the curves indicates that the function relating pipe-wall temperature to the angle on the perimeter is probably of an exponential type. The solid lines in each of these curves has the shape of a parabola. The dotted lines indicate the possibility that the function is not continuous, but that each half of the curve may be a portion of a curve representing a function of higher degree. Since Nusselt's equation is of the fourth degree, it is quite possible that an equation representing these curves would also be of the fourth degree.

Undoubtedly an equation could be derived for the variation of pipe-wall temperature around the circumference of a horizontal pipe. However, this equation would be of no value if it were specific to this one experiment. More data on the condensation of several vapors, condensing on pipes of different diameters, would be necessary in order to derive a general equation. This equation would be valuable in recalculating data taken in the past with a fixed thermocouple installation, provided the location of the thermocouple is known, and in determining the best location of fixed thermocouples in new installations of equipment.

The necessity for knowing the correct mean pipe-wall temperature may be shown by means of the following figures: The value of h for run No. 7 is 280 B.t.u. per hour per square foot per degree F. If the maximum pipe-wall temperature had been used instead of the average temperature, the value would have been 378. If the minimum pipe-wall temperature had been used, the value would have been 205. If some other pipe-wall temperature were used, the value obtained would fall somewhere between the correct value and one of these two extremes. These figures show that it is necessary to use the correct mean tube temperature in order to obtain correct values for heat transfer coefficients.

Suggested additional experiments in Flow of Heat:

Condensation of benzene vapors at rates other than those given and with varying temperature differences.

Condensation of other organic vapors.

Condensation of benzene and other organic vapors on a vertical tube.

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CHAPTER IV

EVAPORATION AND EVAPORATORS

W. L. FAITH[1] and H. McCORMACK[2]

EVAPORATION

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Walker, Lewis, McAdams, and Gilliland, E. R., Principles of Chemical Engineering, 3d Ed., McGraw-Hill, 1937.

The term *evaporation* as used in this chapter refers specifically to the vaporization of water from some solution of which it is one of the component constituents. A very extended consideration of evaporation and evaporators is to be found in *Elements of Chemical Engineering*, by Badger and McCabe.

Only a few of the more important principles and factors relating to evaporation are here presented. The same kind of treatment is accorded the equipment customarily used for this unit operation.

Evaporation is essentially a unit operation in which the flow of heat is of paramount importance and all of the factors relating to the flow of heat have their part in determining the efficiency or lack of efficiency to be secured in evaporation. Reiterating some of these factors they are: transfer from the heating medium which may be condensing steam or some organic liquid with a high boiling point to the retaining wall between the source of heat and the liquid from which evaporation is to occur. Involved in this transfer are, of course, the temperature and velocity of the heating material, the specific heat of this material, and its viscosity.

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The kind of retaining wall and its thickness are minor factors in the transfer here, as in other problems of flow of heat, the major items are transfer through the liquid film on the heating side and transfer through the liquid film on the side toward which heat is flowing.

The factors of importance on the side towards which heat is flowing are boiling point of the liquid, viscosity of the liquid, temperature difference existing between sources of heat and liquid being heated, velocity of the liquid receiving the heat over the retaining wall, and hydrostatic head insofar as it modifies the pressure over the liquid when the evaporation is under vacuum.

Many of the factors enumerated here can be evaluated and calculations made from these values as they have been in the unit operation of Flow of Heat.

Attention should be called, however, to the difficulty of evaluating some of the items necessary for the complete theoretical calculation of the flow of heat. As, for example, the area through which the liquid receiving heat is flowing is many times indefinite or indeterminable. The velocity at which the liquid is flowing is many times indefinite or indeterminable.

It is always possible to determine the total quantity of heat transferred and with most of the component factors calculable, it is possible to set some rather definite values for those factors which are indefinite or indeterminable.

The chemical engineer is interested in evaporation as conducted in steam jacketed kettles, as conducted in open tanks heated with steam coils, and as conducted in vacuum evaporators consisting of from one to six units and heated by steam applied in many different ways.

The customary steam jacketed kettle varies in capacity from one gallon to several hundred gallons. The steam coil applied in an open tank has about the same range in capacity. The vacuum evaporators are customarily rated in square feet of heating surface and in liquid capacity. They may range from laboratory units containing not more than ten square feet of heating surface up to sextuple effects with as much as two thousand square feet of heating surface per effect.

The heating tubes in the vacuum evaporators may be horizontal, vertical, or inclined. The liquid to be evaporated may flow either inside or outside these tubes.

The velocity of the liquid may be attained solely by temperature difference or it may be secured through a circulating pump. Evaporators of the first type would be said to have thermal circulation, while those of the second type would be said to have forced circulation.

The size of tubes, like the heating surface, may vary over a wide range. The smallest is probably one-fourth inch, while the maximum diameter may be two inches. The materials of construction may be almost anything desired. The most common materials are cast iron bodies, ordinary steel tubes, or steel bodies with ordinary steel tubes. Evaporators are, however, built of copper, nickel, aluminum, and stainless steel.

In most industrial processes the source of heat is steam. The heat evolved by condensation of the steam is transmitted through metal walls (of coils, tubes, castings, etc.) and thence to the boiling solution. Evaporation may therefore be considered as a special case of heat transfer, i.e., condensing vapor to a boiling liquid through a retaining wall, and should be treated as such in experimental work.

One would expect, then, that the problem of heat transfer in evaporators could be divided into a determination of the resistances of the individual films concerned, namely, those for condensing steam and for boiling liquid. Unfortunately such is not the case, since data for heat-transfer coefficients from metal to boiling liquid have never been related to a workable general equation. Many factors make the accurate determination of such a coefficient extremely difficult. Among these are the difficulty of measuring the temperature of the film, the variation of the film temperature from point to point on the metal surface, the effect of even minute amounts of scale, and the constant variation of density and viscosity of most boiling solutions.

The accurate determination of the coefficient for condensing steam is not quite so difficult, but doubt often exists as to whether film or dropwise condensation or a combination of the two is taking place.

Consequently, the experiments presented here are arranged primarily as operating experiments, or evaporator tests. On the other hand, this arrangement should not be allowed to obscure the fact that a study of evaporation is essentially a study of heat transmission.

EXPERIMENT NO. 22

HEAT TRANSFER IN JACKETED KETTLES

Practically no information can be found in the technical literature on the subject of heat transfer in jacketed kettles. The lack of dependable experimental work and data is probably accounted for by the difficulties previously mentioned.

Reviewing the formulas for the film coefficients of condensing vapor and for liquid, they are found to be: for vapor condensing on vertical or inclined walls,

$$h = 0.943\left(\frac{k^3\rho^2 gr(\sin\psi)}{N\mu(\Delta t)}\right)^{0.25} \tag{1}$$

A liquid heated by convection in the kettle is most nearly analogous to heat transfer to liquid in a pipe of constantly varying diameter, and the formula which seems most applicable is:

$$\frac{hD}{k} = 0.0225\left(\frac{DG}{\mu}\right)^{0.8}\left(\frac{C_p\mu}{k}\right)^{0.4} \tag{2}$$

The two equations given are probably applicable to the calculation of the quantity of heat transferred in jacketed kettles if it were possible to secure experimental values for the constants in the two equations.

Object. To determine the transfer of heat from a condensing vapor, steam, to a boiling liquid, water.

To calculate the efficiency and steam economy of this process in each type of kettle.

To compare the amount vaporized per unit of time, per unit of area, in kettles of different material and of variable heating space.

Equipment. Cast iron, copper, aluminum, and enameled steel jacketed kettles, either bare or lagged. Tanks and scales for weighing amounts of water fed and steam condensed.

PRELIMINARY REPORT

An outline of the data to be taken and the calculations to be made, together with a description of the procedure to be followed in collecting these data.

Discuss the difficulties involved in securing the constants necessary for the theoretical calculations.

Tabulate, in one group, the ones believed to be determinable, and in another group the ones which seem to be indeterminable.

The assumption of values for two constants will enable the desired calculations to be made. What constants are these?

Assume such values and make the indicated calculations as a part of the final report.

REPORT

This should contain data and calculations equivalent to those listed below as sample data, and the theoretical calculations previously referred to. There should be a brief discussion of the causes leading to the varying results obtained with the different kettles.

All the pertinent data, with calculations for each kettle, should finally be tabulated on a single sheet.

SAMPLE DATA AND CALCULATIONS

HEAT FLOW IN COPPER KETTLE:

Atmospheric pressure	14.5 lbs./in.^{2}
Gage pressure (average)	11.5 lbs./in.^{2}
Total pressure	26.0 lbs./in.^{2}
Steam quality	98%
Steam temperature	242° F.
Weight of water added (water heated)	25.6 lbs.
Weight of water evaporated	30.6 lbs.
Weight of condensate	36.7 lbs.
Average temperature at water added	102° F.
Average temperature of condensate	207° F.
Time of test	1 hr.
Area of heating surface	3.85 ft.^{2}

Heat supplied by steam,

$$\begin{aligned} Q_s &= \text{(lbs. condensate) (latent heat} \times \text{steam quality)} \\ &\quad + \text{(lbs. condensate) (b.p.} - \text{condensate temp.)} \\ Q_s &= 36.7\,(950 \times 0.98) + 36.7\,(242 - 207) = 35{,}487 \text{ B.t.u.} \end{aligned}$$

Heat used in evaporating water,

$$\begin{aligned} Q_w &= \text{(lbs. water evaporated) (latent heat)} \\ &\quad + \text{(lbs. water added) (b.p.} - \text{addition temperature)} \\ Q_w &= 30.6\,(971) + 25.6\,(211 - 102) = 32{,}503 \text{ B.t.u.} \\ \text{Heat lost} &= Q_s - Q_w = 35{,}487 - 32{,}540 = 2947 \text{ B.t.u.} \end{aligned}$$

Overall coefficient, B.t.u./(ft.)^2 (° F.) (hr.)

$$= Q_w/\text{time in hrs.} \times 1/\text{area in ft.}^2 \times 1/\Delta t \text{ (steam and b.p. of water)}$$

$$= \frac{32{,}503}{3.85\,(242 - 211)} = 272$$

Efficiency of heat transfer, $Q_w/Q_s \times 100 = \dfrac{32{,}450}{35{,}487} \times 100 = 91.6\%$

Steam economy, lbs. water evaporated per lb. steam $= \dfrac{30.6}{36.7} = 0.835$

Lbs. water evaporated per ft.^{2} per hr. per ° F. $= \dfrac{30.6}{3.85 \times 1 \times 31} = 0.256$

VACUUM EVAPORATION

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Most industrial evaporation processes make use of vacuum for one of two reasons, either to lower the boiling point of a solution containing a heat-sensitive substance, or to effect an economy of steam.

In either case, several types of equipment are available. The variations between different pieces of apparatus are chiefly in the method of heating employed. For instance, the steam may be in a jacket, as in the common vacuum pan, inside a coil, as in certain vacuum pans used in the sugar industry, or on either side of tubes, as in standard evaporator types; the tubes mentioned may be horizontal, vertical, or inclined; the liquor may be circulated by natural or forced convection. Finally the evaporation may be carried out in single effect with either live or exhaust steam, or in multiple effect where the steam pressure varies from one effect to the other.

Two experiments are presented for a study of vacuum evaporation. These experiments, like those on the jacketed kettles, are essentially experiments in flow of heat. But, again, certain indeterminate conditions of operation make it impossible to, *a priori*, calculate the film coefficients at the transfer surfaces. The experiments are therefore arranged primarily as operating experiments.

EXPERIMENT NO. 23

TEST OF A SINGLE-EFFECT EVAPORATOR (BATCH OPERATION)

Object. 1. To determine the heat necessary to raise the temperature of the apparatus to that necessary for evaporation at atmospheric pressure.

2. To determine the efficiency of heat transfer of the evaporator when operated at atmospheric pressure. To make a condenser balance.

3. To determine the heat given up by the apparatus when changing from atmospheric pressure to vacuum.

4. To determine the efficiency of heat transfer of the evaporator when operated under vacuum, and to make a check condenser balance.

Equipment. Any single-effect vacuum evaporating apparatus equipped with a surface condenser. In many laboratories one effect of a multiple-effect evaporator may be used. If a jet condenser only is available, Parts I and III must be omitted. The size of the evaporator should be such that it will hold at least 100 lbs. of water. An evaporator, with the heating arrangement shown in Figs. 1, 2, and 3 has been found very satisfactory.

Procedure. Read the references listed, particularly No. 3. Then find out as completely as possible about the design and method of operation of the apparatus to be used.

This test is composed of four parts, and is so arranged that each part follows the other with no time interval between successive parts.

In Part I, sufficient data should be taken to calculate the amount of heat necessary to raise the temperature of the apparatus to the point where evaporation begins. Part I ends and Part II begins when the first few drops of condensate come from the condenser.

The data necessary for Part II should then be taken. In order to get a good condenser balance, temperatures should be read often and accurately. Why? 20 to 30 lbs. of distillate should be collected during this test.

Part II is concluded by:

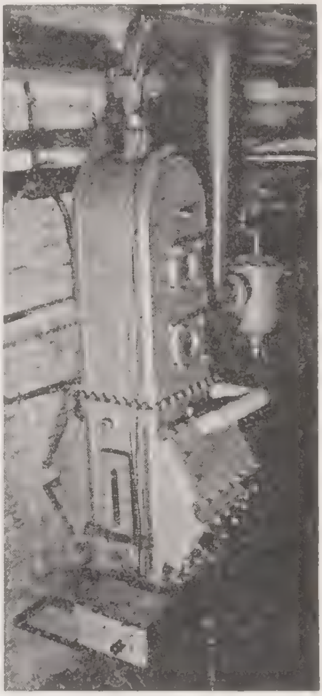

Fig. 1.—Cross tube evaporator.

1. Turning off steam supply.
2. Exchanging receivers of distillate after flow stops.
3. Concluding condenser temperature readings when flow stops, and recording final water meter reading.

Part III begins when the vacuum is turned on, and ends when no more distillate comes over, and vacuum readings have reached a constant value.

In Part IV, 20 to 30 lbs. of distillate should be collected. Approximately 30 lbs. of water should remain in the evaporator at the end of the test.

Note: The various quantities listed under this procedure are for an initial feed charge of 100 lbs. of water. If the evaporator to be tested requires a greater charge than this, the quantities listed above should be increased proportionally.

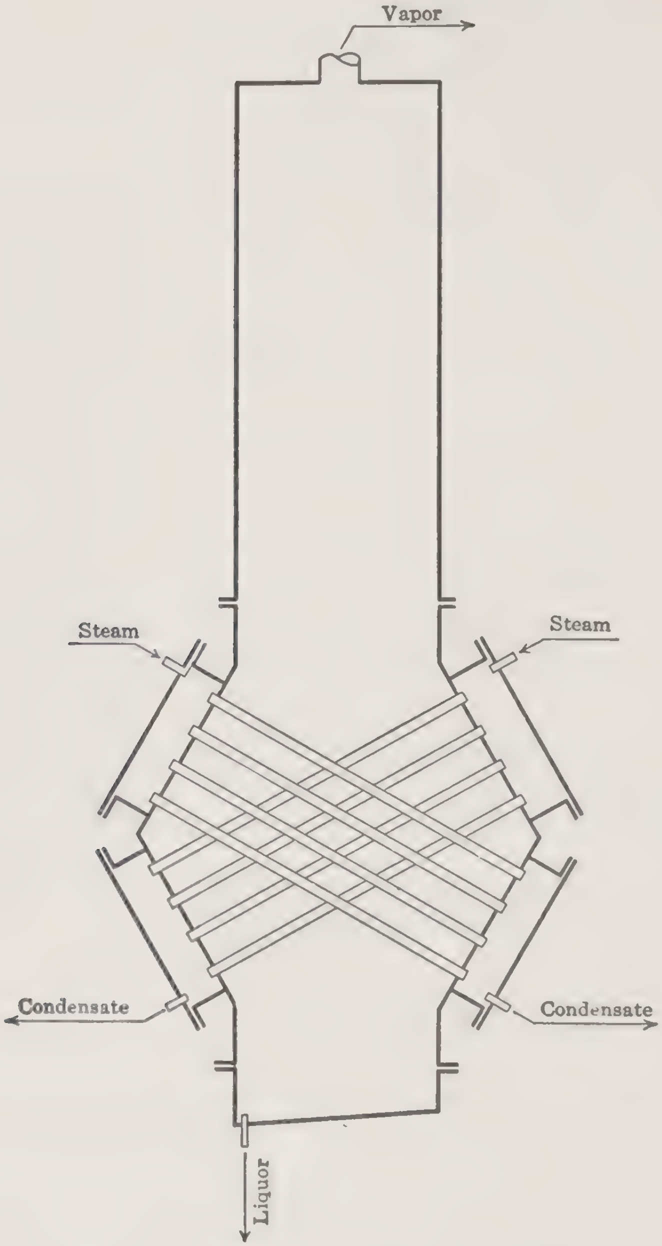

FIG. 2.—Diagram of cross tube evaporator.

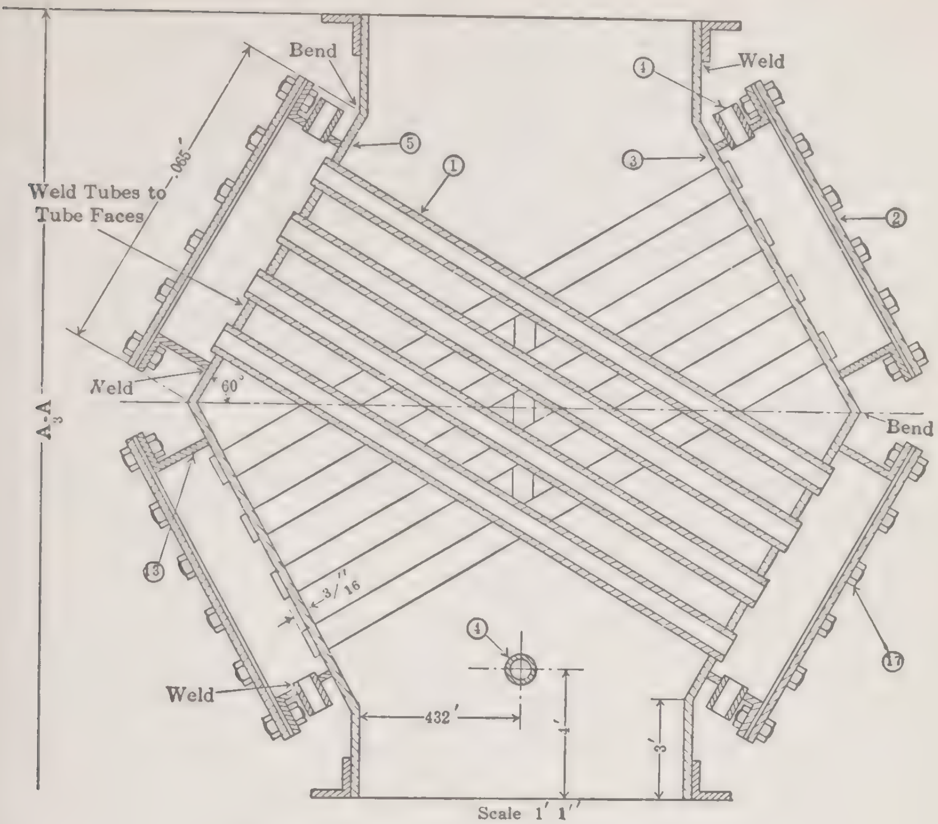

Fig. 3.—Tube and steam chart arrangement.

PRELIMINARY REPORT

Describe the exact procedure of the test, the data to be determined, and the method of so doing. Answer the following questions:

1. Determine the amount of distillate leaving through the vacuum pump. If this information cannot be obtained, how apportion this loss in the calculations?

2. In Part III, what may be expected as to the comparative rates of cooling the water and the evaporator body? How would this discrepancy affect the calculations?

3. Answer the question asked under procedure.

Discuss briefly the effect of the following factors in evaporation processes: (*a*) non-condensable gases in the steam chest, (*b*) hydrostatic head, (*c*) scale formation, (*d*) quality of steam, (*e*) entrainment, (*f*) liquid viscosity.

REPORT

Make the calculations indicated under Purpose, together with those shown in the sample data and calculations.

Discuss the effect of the various factors studied on a commercial evaporation process. Compare the two condenser balances and discuss any discrepancies observed. Compare results obtained during atmospheric evaporation with those of vacuum evaporation.

List 10 manufacturers of commercial evaporators.

SAMPLE DATA AND CALCULATIONS

Part I

Weight of feed water	100.8 lbs.
Temperature of feed water	70° F.
Steam pressure	20.0 lbs. gage
Barometer, 740 mm	14.3 lbs./in.^{2}
Quality of steam	85.6%
Weight of condensate	29.44 lbs.
Temperature of condensate	161° F.

Heat given up by steam:

$$29.44\ (258.1 - 161.0) + 29.44\ (.856 \times 939.6) = 2860 + 23{,}650 = 26{,}510 \text{ B.t.u.}$$

Heat necessary to raise contained water to distillation:

Sp h · Heat $$100.8\ (210.6 - 70.0) = 14{,}120 \text{ B.t.u.}$$

Part II

Weight of steam condensate	54.3 lbs.
Temperature of steam condensate	210° F.
Weight of distillate from condenser	39.9 lbs.
Temp. distillate from condenser	73.4° F.

Heat required to evaporate distillate: $39.9 \times 971.5 = 38{,}800$ B.t.u.

Heat given up by steam:

$$54.3\ (258.1 - 210) + 54.3\ (.856 \times 939.6) = 2615 + 43{,}600 = 46{,}215 \text{ B.t.u.}$$

Efficiency of heat transfer (operation at atmospheric pressure):

$$\frac{38{,}800}{46{,}215} \times 100 = 84.1\%$$

Condenser balance:

Volume of cooling water	125.8 cu. ft.
Temperature of water entering condenser (average)	57.6° F.
Temperature of water leaving condenser (average)	63.1° F.

Heat given up by condensing vapor: $38{,}800 + 39.9\,(210.6 - 73.4) = 44{,}280$ B.t.u.
Heat carried away by condenser water: $125.8 \times 62.3 \times 5.5 = 43{,}050$ B.t.u.

$$\text{Error in condenser balance: } \frac{44{,}280 - 43{,}050}{44{,}280} \times 100 = 2.78\%$$

Part III

Weight of distillate collected	8.8 lbs.
Temperature of condenser body at end of run	74.0° C.
Vacuum at end of run	26.7 in. Hg

$$\text{Average latent heat of water during run: } \frac{971.5 + 1031.5}{2} = 1001.5$$

Heat required by water evaporated: $8.8 \times 1001.5 = 8813$ B.t.u.
Heat given up by cooling feed water: $(100.8 - 39.9)\,(210.6 - 108) = 6240$ B.t.u.
Heat given up by evaporator body and by radiation: $8813 - 6240 = 2573$ B.t.u.

Part IV

Weight of steam condensate	61.3 lbs.
Temperature of steam condensate	210° F.
Weight of distillate from condenser	42.6 lbs.
Temperature of distillate from condenser	107.5° F.
Average vacuum	23.5 in. Hg
Weight of water withdrawn at end of test	4.7 lbs.

Heat given up by steam:
$61.3\,(258.1 - 210) + 61.3\,(.856 \times 939.6) = 2945 + 49{,}200 = 52{,}145$ B.t.u.

Material balance:

Withdrawn at end	4.7 lbs.
Distillate Part IV	42.6 lbs.
Distillate Part III	8.8 lbs.
Distillate Part II	39.9 lbs.
Total accounted for	96.0 lbs.

Feed water unaccounted for or passing through vacuum pump:

$$100.8 - 96.0 = 4.8 \text{ lbs.}$$

∴ Weight of water evaporated: $4.8 + 42.6 = 47.4$ lbs.

$$\text{Absolute pressure in evaporator: } 14.3 - \frac{23.5}{29.92} \times 14.7 = 2.75 \text{ lbs. abs.}$$

Heat required to evaporate water = heat to raise feed to 137.7° F. + heat to evaporate water at 137.7° F. $= 52.1\,(137.7 - 108) + (47.4 \times 1015) = 1550 + 48{,}000 = 49{,}550$ B.t.u.

$$\text{Efficiency of heat transfer} = \frac{49{,}550}{52{,}145} \times 100 = 95\%$$

Condenser balance:

Volume of cooling water	130.0 cu. ft.
Temperature of inlet water	57.6° F.
Temperature of outlet water	63.0° F.

Heat given up by vapor condensed:

$$42.6\ (1015) + 42.6\ (137.7 - 107.5) = 43{,}200 + 1288 = 44{,}488 \text{ B.t.u.}$$

Heat carried away by condenser water: $130 \times 62.3 \times 5.4 = 43{,}700$ B.t.u.

$$\text{Error in condenser balance} = \frac{44{,}488 - 43{,}700}{44{,}488} \times 100 = 1.77\%$$

EXPERIMENT NO. 24

TEST OF A MULTIPLE-EFFECT EVAPORATOR

Object. A study of the conditions for the satisfactory operation of a multiple-effect evaporator, and determination of its operation characteristics.

Tabulation of operating conditions and operating results, showing efficiency of heat transfer and apparent overall heat-transfer coefficients for each effect; steam economy and capacity of entire system.

Procedure. Inspect the equipment and its accessories. Trace the course followed through the evaporator by the feed, exhaust steam, first-effect vapor and second-effect vapor. Note where the various thermometers and gauges are located, and determine the utility of each.

Make provision for measuring all variables necessary for the data and calculations.

Search the apparatus for leaks, and repair any which are found.

Consult one of the instructors for steam pressure and vacuum to be maintained.

PRELIMINARY REPORT

Describe the exact procedure of the proposed test. Then describe briefly the principles of multiple-effect evaporation. Discuss briefly (*a*) methods of feeding, (*b*) condensate removal, (*c*) control of rate of evaporation, (*d*) procedure in starting and stopping a multiple-effect evaporator, (*e*) removal of air from steam chests. Describe exact procedure of test.

Indicate the variables entering into a heat balance of (*a*) first effect, (*b*) second effect, (*c*) third effect of an evaporator. Compare the economy and capacity of two single-effect evaporators and one double-effect evaporator made up of the same units and operated with the same total vacuum.

Distinguish between forward feed, backward feed, and parallel feed. State the advantages and disadvantages of each.

REPORT

Make the tabulations and calculations indicated under Object together with those shown in the Sample Data and Calculations. Discuss your operation of the equipment and suggest any changes in methods or in the equipment set-up which would lead to more satisfactory operation.

CHAPTER V

DISTILLATION

JOSEPH H. KOFFOLT[1] and JAMES R. WITHROW[2]

Distillation is a chemical engineering operation used in meeting the engineering requirements of separation of liquids from liquids, liquids from solids, and gases from liquids, the operation used to meet the latter requirement, however, is sometimes also termed stripping, stabilizing, or desorption.

The operation in its more complex form involves a series of engineering steps, as: storage, preheating, charging, heating, vaporizing, condensing, fractionating, cooling, with heat exchange and recovery of the final product as distillate. The degree of separation desired of the components from a mixture, and hence the particular engineering equipment to be used as: simple still, simple continuous still (*flash or equilibrium still*), dephlegmating still, live steam still, and column still is determined by the demands of the market and economics, the chemistry of the process, and other engineering requirements.

The vapor-liquid phase equilibria relationships between the components constitute the fundamental and basic data for most distillation problems. Only two component systems (binary mixtures) will be considered in this discussion.

EQUILIBRIUM DIAGRAM

Underlying Principles. It has been found that the resulting vapor pressure of a solution of two liquids miscible in all proportions or to any extent miscible in one another is less than the vapor pressures of the two liquids before mixing. In these cases the resulting vapor composition has been found to be dependent upon many factors, as, the relative amounts of each in solution, the nature and properties of the liquids in solution, and the total pressure of the system.

The vapor-liquid equilibria relationships of solutions which closely approximate the properties of an *ideal* or *perfect* solution as, homologues of a series of non polar organic compounds (for example, benzol and toluol, some substances like paraffin hydrocarbons) may be calculated by a generalization known as *Raoult's Law.* This generalization states that at any particular temperature the equilibrium vapor pressure exerted by a component in solution is equal to the mol fraction of that component in solution multiplied by its vapor pressure in the pure state as liquid at this temperature. By application of Dalton's Law and Avogadro's hypothesis, the vapor-liquid equilibrium relationship for any particular temperature may be calculated. Examination of vapor-liquid equilibria diagrams (Fig. 1) and data [1] indicate that most of the binary mixtures do not even approximate Raoult's Law, the vapor-liquid equilibria relationships must therefore be determined in practically all cases by experimental means. There are many methods in the literature for the experimental determination of vapor-liquid equilibria relationships; as the method of Carveth,[2] Petit,[3] Rosanoff, Bacon and White,[4] Carey and Lewis,[5] Scatchard,[6] and others.

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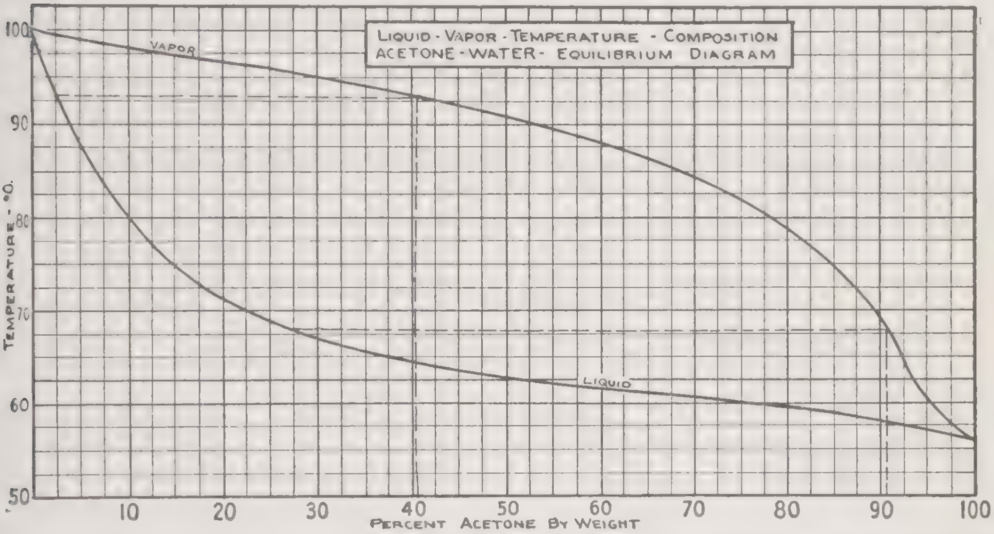

FIG. 1.—Liquid-vapor-temperature-composition. Acetone-water-equilibrium diagram.

[1] Carey, Chemical Engineers Handbook. Edited by John H. Perry, by McGraw-Hill Book Company. Section 12, Distillation, page 1105 et seq. 1934.

Badger and McCabe, Elements of Chemical Engineering. McGraw-Hill Book Company. 2nd Edition, page 323 et seq. 1936.

Walker, Lewis, McAdams, and Gilliland, Principles of Chemical Engr. McGraw-Hill Book Co., 3rd Edition, page 514 et seq. 1937.

[2] Carveth, The Composition of Mixed Vapors. Journal Physical Chemistry 3, 193 (1899).

[3] J. H. Petit, Minimum Boiling Point and Vapor Composition. Journal Physical Chemistry 3, 360 (1899).

[4] Rosanoff, Bacon, and White, Journal American Chemical Society 36, 1803 (1914).

[5] Carey and Lewis, Studies in Distillation. Industrial Engineering Chemistry 24, 882 (1935).

[6] Scatchard, Journal American Chemical Society 60, 1275 1278 (1938).

The objections to many of the experimental methods may be summarized: (1) inaccuracies inherent in the methods; as, fractionation of the first vapors evolved, dephlegmation due to heat losses during the course of the experiment, relationships determined over a range of temperatures, and: (2) complexity of set up and procedure.

A highly convenient apparatus for this type of determination is the Othmer apparatus,[1] as designed and modified by E. K. Smoley and described by Carey and Lewis.[2] This determination of vapor-liquid equilibria relationships consisted of continuous recycling of condensed vapors (distillate) to the equilibrium still. Thus when steady conditions were established by observing that refluxing back to the still had ceased and that there was no change in vapor temperature, the composition of distillate returning to the still was then assumed to be the same as the composition of vapors leaving the still. Then by analysis of the distillate and of the liquid in the still, for various runs, the vapor-liquid equilibria relationships were determined.

SIMPLE BATCH DISTILLATION

Simple batch distillation is one of the methods of meeting the engineering requirement of partial separation of the more volatile components from a mixture of liquids by charging into a still and heating. The vapors evolved, by boiling the liquid mixture, are delivered continuously from the still as produced, condensed, and collected in receivers without allowing any pre-arranged condensation or refluxing of the condensate to the still to take place. In boiling such a liquid mixture the composition of the vapors at any instant are assumed to be in equilibrium with the composition of the liquid in the still. The vapors evolved will be richest in that component which has the highest partial vapor pressure for the mixture being distilled.

The principle underlying simple batch distillation can best be illustrated by reference to a vapor-liquid equilibrium boiling point diagram, and as shown in Fig. 1. Let it be assumed that a mixture of 27.5% acetone in water is charged into the kettle of a simple batch still, and heat is applied. The mixture will first start to boil as indicated on the diagram (Fig. 1) at a temperature of 68° C. The composition of the vapors evolved, which are in equilibrium with the composition of the liquid in the still, from Fig. 1, will be 91% acetone in water. As distillation proceeds and vapors are continuously removed and condensed, there will result: a progressive decrease in the quantity of liquid in the still, and of the composition of the more volatile component in the liquid and the vapor, and a progressive increase of the boiling temperature of the liquid in the still. For example, when the composition of the liquid in the still has decreased from 27.5% to 2.5% acetone the composition of the vapors (Fig. 1) will have decreased from 91 to 40.5% acetone while the boiling temperature of the liquid in the still will have risen from 68° to 93° C.

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[1] Othmer, Composition of Vapors from Boiling Binary Solutions. Industrial Engineering Chemistry 20, 743 (1928).

[2] loc. cit.

The computations involved in predicting the composition and quantity of distillate from simple batch distillation are therefore differential in character, and require for their solution the calculus. Lord Rayleigh[1] derived the following equation for simple batch distillation of binary mixtures:

$$2.3 \log \frac{W_0}{W_r} = \int_{x_r}^{x_0} \frac{dx}{y - x} \tag{1}$$

where W_0 = Weight of charge whose composition is $x_0\%$ by weight of more volatile component at the beginning of simple distillation

W_r = Weight of residue of composition $x_r\%$ by weight of the more volatile in the still at end of distillation

y = Composition of the vapor in equilibrium with the liquid of composition (x) in the still, % by weight

The quantity $\int_{x_r}^{x_0} dx/y - x$ may be integrated directly if "y" can be expressed in terms of "x", as in the special case where Raoult's or Henry's Law apply,[2] or between ranges of compositions where an approximately straight line relationship exists between x and y.[3]

The general and more simple method of evaluating this integral is by the graphical integration method where "x" is plotted versus $1/y - x$ and the area underneath the curve between the limits of "x_0" and "x_r" is measured by means of a polar planimeter or computed by one of the graphical integration methods.[4] The former method is illustrated later in this section.

The composition of the distillate (x_d) (% by wt.) in terms of the more volatile component may be computed from the following equation:

$$x_d = \frac{(W_0 x_0 - W_r x_r) \times 100}{(W_0 - W_r)} \tag{2}$$

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[1] Lord Rayleigh. Phil. Mag. VI, 4, 521 (1902).

[2] Walker, Lewis, McAdams, and Gilliland, ibid., page 532. Badger and McCabe, ibid., page 335. Carey, ibid., page 1153.

[3] Robinson and Gilliland, The Elements of Fractional Distillation. McGraw-Hill Book Co., 1939. 3rd Edition. Pages 48 and 50.

[4] Sherwood and Reid, Applied Mathematics in Chemical Engineering. McGraw-Hill Book Co. (1939).

SIMPLE CONTINUOUS DISTILLATION

Simple continuous distillation (equilibrium or flash distillation) may also be used in meeting the engineering requirement for the partial separation of the more volatile components from a mixture of liquids. It consists in operating the still continuously under constant conditions, of rate and compositions, with the partial vaporization of feed stock and withdrawal of residue from the still in such a manner that equilibrium exist between the liquid and vapors in and leaving the still. This method of distillation finds extensive application in petroleum refining as in "pipe stills."[1]

Simple continuous distillation is distinguished from simple batch distillation in that it is continuous in operation; constant conditions of composition and rate of feed, distillate, and residual liquid prevail at all times; and permits a greater percentage of material to be vaporized at a given temperature than simple batch distillation,[2] but will contain less of the more volatile component.

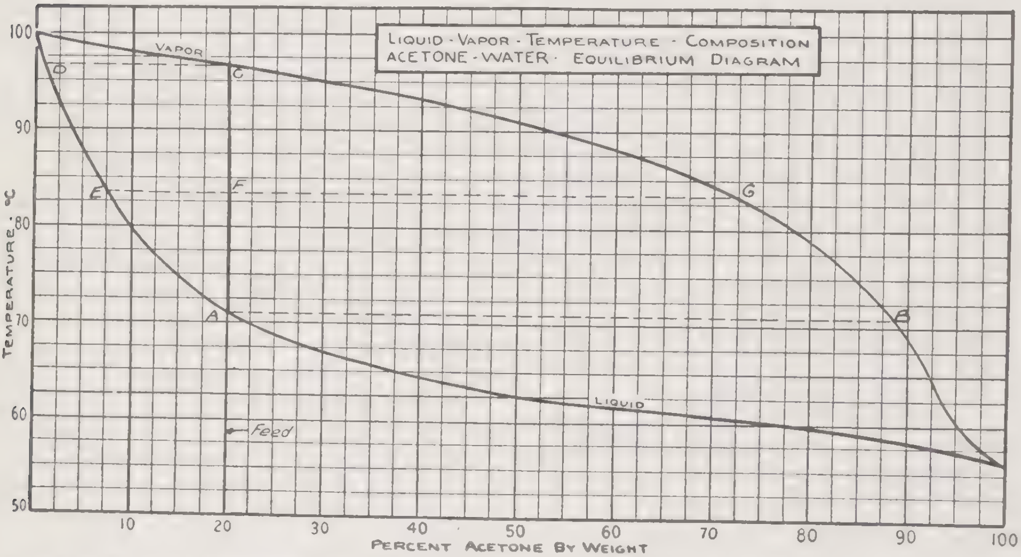

FIG. 2.—Liquid-vapor-temperature composition. Acetone-water-equilibrium diagram. (Simple continuous distillation).

The principle underlying simple continuous distillation can best be illustrated by reference to a vapor-liquid equilibrium diagram, and as shown in Fig. 2.

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[1] Piroomov and Loomis, Design Problems of Flash Distillation Equipment in the Petroleum Industry. Trans. Amer. Inst. Chem. Eng., *22*, 55 (1929).

Nelson, Petroleum Refinery Engineering. McGraw-Hill Book Co., page 238 (1936).

[2] Carey, Chemical Engineers Handbook, ibid., page 1173.

Let it be assumed that unit weight of a mixture containing 20% acetone in water be fed to a simple continuous still and an *infinitesimal* amount be *vaporized*, the remainder being withdrawn from the still as residue. Since equilibrium conditions are assumed to prevail the composition of this *infinitesimal* amount of vapor is as indicated on the diagram, to be approximately 88% acetone in water, while the composition of the residue is practically the same as the composition of the feed, namely, 20% acetone in water, the temperature at which this distillation is carried out is 71° C. The tie line AB on this diagram may also be used to represent the amount of feed, since the amount of feed vaporized was infinitesimal and is therefore negligible. The line of AB for this particular case also represents the amount of residual liquid withdrawn from the still. It will be noted also that the line AB which represents the amount of liquid withdrawn lies to the *right* of the feed line AC.

Now let it be assumed that the great bulk of the feed to the still is vaporized and an *infinitesimal* amount is withdrawn as *residue*. Similarly, the composition of the vapor will be practically the same as the composition of the feed, 20% acetone in water, while the composition of the residue, which is in equilibrium with the composition of the distillate vapors is 0.75% acetone in water, the temperature of distillation for this particular case is 97° C. Since the amount of residual liquid withdrawn from the still can be assumed to be negligible, the line CD represents the amount of feed, and the amount vaporized. It will be noted that the line CD lies to the left of the feed line AC.

The above illustrations represent conditions of maximum and minimum vaporization of materials fed to the still. It will be noted that the temperature of distillation may vary from 71° C. to 97° C.; composition of distillate from 20% to 88% acetone in water, and the composition of residual liquid from 0.75% to 20% acetone in water.

Let it now be assumed that the distillation is conducted under such conditions that a vapor containing 72.5% acetone in water, represented by point G on the diagram is desired. The equilibrium tie line, EG, will therefore represent the following in regard to composition and quantities; point F, composition of the feed; point G, the composition of vapor (or distillate); point E, composition of residual liquid (7.5% acetone in water); the line EG, the quantity of feed; FG, the quantity of residual liquid withdrawn from the still; the line EF the quantity of vapors (or distillate) from the still; and finally the intersection of line EG with the ordinate, the temperature of distillation namely, 83.5° C. Therefore,

$$\%\ \text{Vaporized} = \frac{EF}{EG} = \frac{(20 - 7.5)100}{(72.5 - 7.5)} = 19.3\% \qquad (3)$$

$$\%\ \text{Liquid withdrawn as residue} = \frac{FG}{EG} = \frac{(72.5 - 20)100}{(72.5 - 7.5)} = 80.7\% \qquad (4)$$

The above method of calculation by inverse proportions is sometimes known by the hybrid name "Lever-Reaction." The preceding illustrations may be calculated also from a material balance equation.[1]

$$Fx_f = Vy + (F - V)x_w \tag{5}$$

Where F = Weight of feed of composition x_f
V = Weight of feed vaporized
y = Composition of vapors in equilibrium with liquid of composition x_w withdrawn from the still

VAPOR ENRICHMENT BY PARTIAL CONDENSATION (DEPHLEGMATION)

It has been shown in the previous discussion that the more volatile components of a given mixture subjected to either simple batch or simple continuous distillation are obtained in increased concentration in the vapor as compared with that simultaneously in the still, the proportions of which are determined by liquid-vapor equilibria phase relationships. The composition of the distillate, however, was the same as the composition of the vapors evolved in the still.

The demands of the market and economics, the chemistry of the process, and other engineering requirements quite often require a higher concentration of distillate in the more volatile components than can be obtained in one stage by either simple batch or simple continuous distillation. These demands, however, can be met by various engineering means as: repeated simple distillations, (usually prohibitive from the standpoint of high heat consumption, and high operating and maintenance cost); vapor enrichment by partial condensation (dephlegmation) of vapors evolved from a simple distillation resulting in an enrichment of the more volatile constituents of the vapors; and column still distillation.

Vapor enrichment by partial condensation (dephlegmation) consists in the partial condensation of a mixture of vapors by cooling them through contact with a cooled solid surface, (for example, a tubular condenser), to a final definite temperature, the composition of the condensate being weaker in the more volatile components than the original vapor mixture while the composition of the uncondensed vapors (which eventually becomes the distillate) is stronger. The composition of the condensate and the uncondensed vapors are assumed to be in equilibrium with one another and are dependent upon the degree of cooling of a particular mixture.[2]

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[1] Walker, Lewis, McAdams, and Gilliland, ibid., page 532. Badger and McCabe, ibid., page 335.

[2] Condensate refers to the mixture of vapors condensed in the partial condenser (dephlegmator) and returned to the still kettle, while the uncondensed vapors are termed distillate after condensation in the final condenser and collection in receivers.

At the present time, partial condensers (dephlegmators) find somewhat limited use in connection with simple distillation as the demands of a more concentrated distillate, in terms of the more volatile component, can usually be more advantageously met by the more efficient method of column still distillation,[1] though this equipment is more expensive.

Partial condensers are, however, sometimes used in place of reflux condensers in column stills (light oil and benzol refining) and especially with vacuum stills in petroleum refining.[2]

Partial condensers (dephlegmators) differ from reflux condensers, in that a practical enrichment of the vapor is accomplished, while in the latter type of equipment the sole function is to furnish reflux liquid for the column with no, or, negligible, enrichment of the vapors taking place.

Partial condensation of the vapors from a distillation operation may be produced in two ways: *simple, partial condensation*, in which the condensate formed is kept in contact and equilibrium with the vapors throughout the whole condensing period; and *differential partial condensation*, in which the condensate is continuously removed from contact with the residual vapor as fast as it is formed.

The underlying principles and mathematical treatment of simple partial condensation is analogous and similar to simple continuous distillation. Fig. 3 illustrates these principles graphically. Let it be assumed that a mixture containing 7.5% acetone by weight is distilled, if equilibrium conditions exist the composition of the vapors will be 72.5% acetone by weight. If these vapors are cooled in a simple partial condenser to a temperature, say 68° C., partial condensation will take place giving a liquid condensate of 28% acetone by weight which is returned to the still, and a residual vapor of 90.5% acetone by weight which is condensed and collected as distillate. From the principle of inverse proportions (so-called "lever reaction")

$$\frac{FG}{EF} = \frac{\text{Relative amount of Vapor Condensed in Partial Condenser}}{\text{Relative amount of Residual Vapor (Distillate)}} \tag{6}$$

or

$$\frac{FG}{EG} \times 100 = \text{Percentage of Vapors Condensed} \tag{7}$$

The amount of condensate can also be calculated from a material balance equation [3]

$$\frac{V_1 - V_2}{V_2} = \frac{y_2 - y_1}{y_1 - x_2} \tag{8}$$

[1] Leslie, Motor Fuels. The Chemical Catalog Co., page 120 (1923).

[2] Nelson, Petroleum Refinery Engr. McGraw-Hill Book Co. pages 397-402 (1936).

[3] Walker, Lewis, McAdams, and Gilliland, ibid., page 534.

where V_1 = Mols of vapor of composition y_1 (mol fraction) to be enriched by simple partial condensation to a composition of y_2 (mol fraction)

V_2 = Mols of residual vapor of composition y_2 (mol fraction)

x_2 = Composition of condensate (partial condensate, mol fraction)

If these vapors were cooled to a temperature of approximately 61° C. so that there remained only an infinitesimal amount of vapor it can be seen from Fig. 3 that the resulting composition of the distillate would be approximately 94.75% acetone by weight. Therefore, by simple partial condensation, it is possible in the particular case discussed to enrich the vapors from a composition of 72.5% to 94.75% acetone by weight, in the latter case, however, only a negligible amount of product as distillate would be obtained.

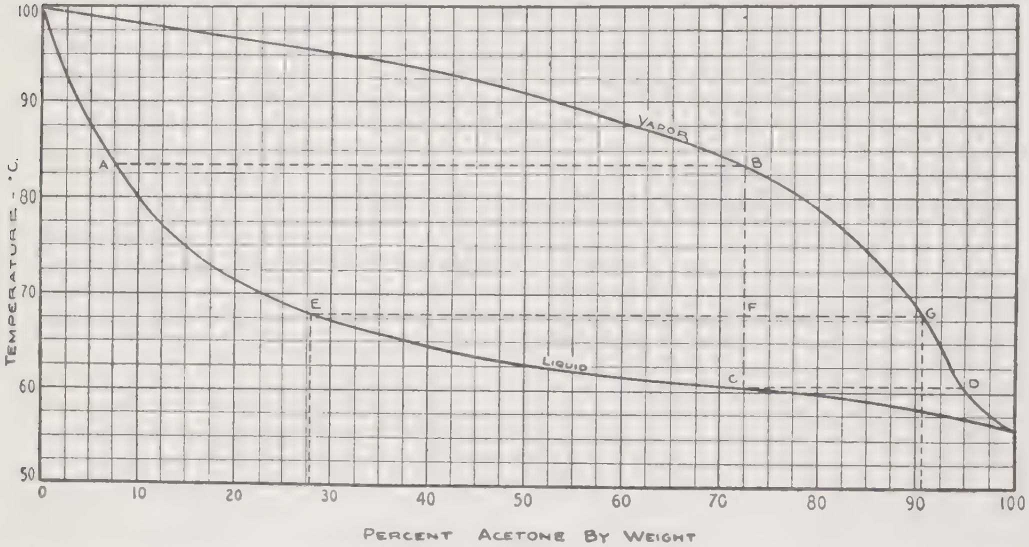

FIG. 3.—Liquid-vapor-temperature composition. Acetone-water equilibrium diagram. (Simple partial condensation).

The mathematical treatment of differential partial condensation is similar to the reasoning followed in the development of the Lord Rayleigh equation for simple batch distillation. Differential partial condensation, in the last analysis, consists of a series of infinitesimal simple partial condensations with a series of infinitesimal increases in composition of the partial condensate and residual vapor in the more volatile component.

Calculations must therefore involve the calculus, the general equation relating the original and residual weight of vapor may be written[1]

$$2.3 \log \frac{V_1}{V_2} = \int_{y_1}^{y_2} \frac{dy}{y - x} \tag{9}$$

where V_1 = weight of vapor of composition y_1 (% by weight) of more volatile component being enriched to y_2 (% by weight) by differential partial condensation

V_2 = weight of residual vapor of composition y_2 (% by weight) of more volatile component collected as distillate

y = composition of the vapor in equilibrium with the partial condensate of composition x (% by weight) of more volatile component in the condenser

BATCH COLUMN STILL DISTILLATION

The engineering requirements are often further complicated by stipulating, in addition to a very high concentration of one of the components in the product, that a greater economy in steam, maintenance, equipment, and operating costs be effected than can be obtained by any of the methods discussed heretofore. These demands may be met by use of the engineering unit known as a column still which may be either intermittent or continuous in operation. The intermittent or batch column still will be considered in this discussion.

A column still distillation unit is shown in Fig. 4. It consists essentially of *a still kettle*, *a rectifying column*, *a condenser* and *a receiver.* The material to be distilled is charged into the *still kettle,* which is equipped with a steam coil for heating and generating vapors, which pass into the column, and a "reflux to still" condensate line for returning liquid (reflux) as overflow, from the bottom plate of the column to the still kettle.

The *rectifying column* which may be either packed with inert materials as, for example, Raschig rings or Berl saddles, or contain a series of plates with provisions on each plate for bringing a continuous stream of vapor from the plate below (in the case of the bottom plate, the still) into intimate contact with the liquid on the plate above which is fed continuously by liquid (reflux), as overflow, from the plate above (in the case of the top plate, from the combination reflux and final condenser). The flow of vapor and liquid in the column are therefore counter-current with respect to one another.

Intimate contact of these two counter-current streams and the liquid on the plate may be obtained by the use of the inert packing material, perforated (sieve) plates, or by bubble caps. The column shown in Fig. 4 contains 14 plates with 4 bubble caps per plate. In this discussion

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[1] Walker, Lewis, McAdams, and Gilliland, ibid., page 534.

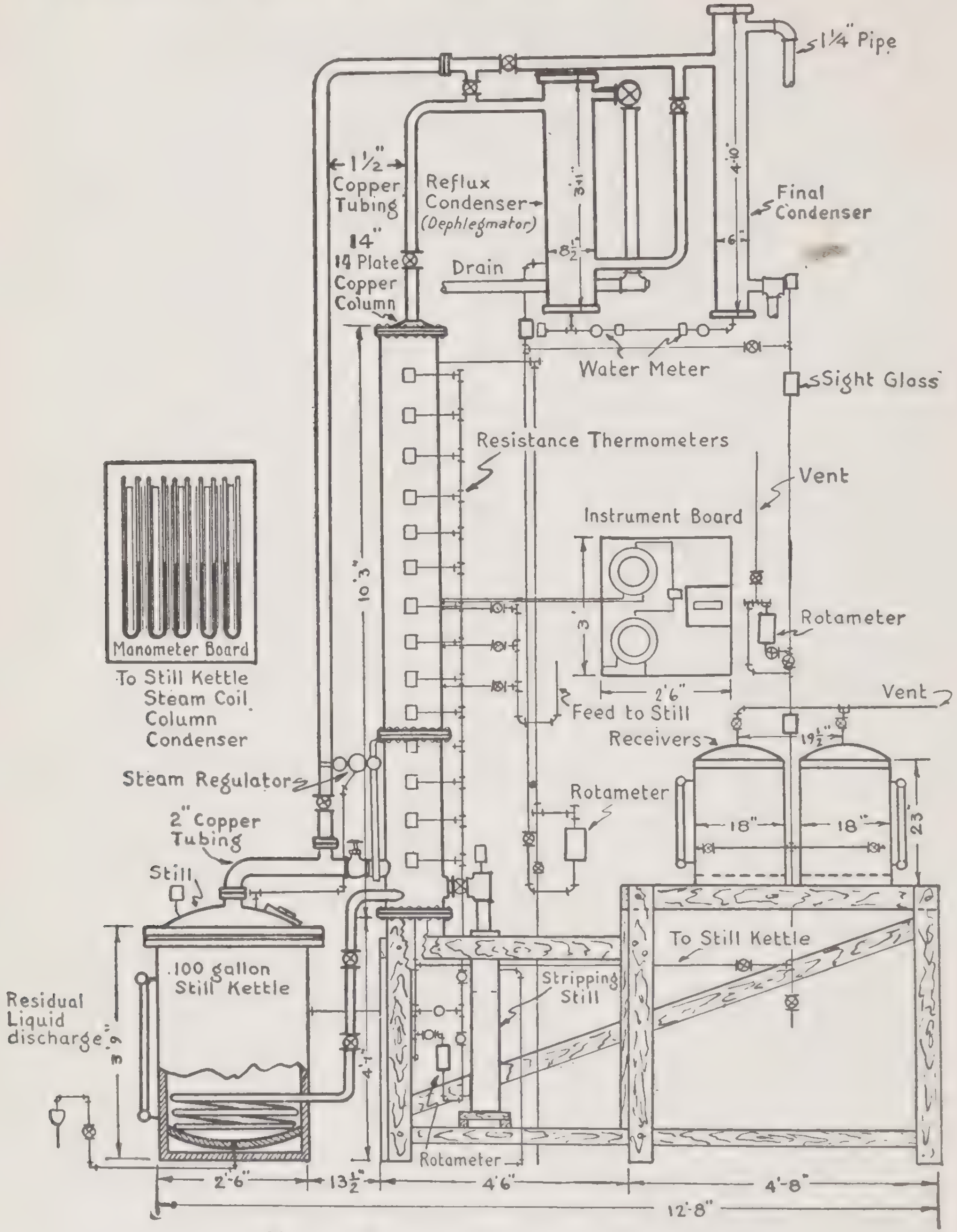

FIG. 4.—Fractional Distillation Unit.

the plates will be numbered from the top down; plate No. 1 being the top plate, and plate No. 14, the bottom plate.

An interchange of heat and transfer of material is obtained by this intimate contacting, resulting in an enrichment of vapor on each succeeding plate proceeding upward. This operation is known as *rectification.* The vapors from the top plate pass into the *combination condenser* where they are condensed and cooled, part of the liquid being returned to the column as reflux and the other part collected in the *receiver,* as product or distillate.

The degree of enrichment in the vapors (and hence the liquid) from plate to plate in the column is dependent upon the vapor-liquid equilibria relationships of the material being distilled; the composition of the product desired; and the reflux ratio (ratio of quantity of reflux to quantity of vapor at any given point in the rectifying column).

There are many methods available for calculating the plate to plate change in composition of vapor and liquid in the rectifying column.[1]

Underwood[2] has summarized most of the existing methods of calculations, Brown[3] gives numerical examples of some of these methods. A highly convenient method is that of McCabe and Thiele,[4] in which the change of composition from plate to plate and also the number of ideal plates required for the separation of binary mixtures, may be computed graphically or algebraically by an equation relating the composition of the vapor from the plate below, y_{n-1}, and the composition of the liquid on the plate above, x_n. This equation is known as the equation of the "operating line" and may be written

$$y_{n-1} = \frac{Rx_n}{V} + \frac{Dx_d}{V} \tag{10}$$

where y_{n-1} = mol fraction of more volatile component in vapor leaving plate "n − 1" (the plate below), and entering plate "n" (the plate above)

x_n = mol fraction of more volatile component in liquid on plate "n"

R = mols of reflux from plate to plate per unit time

V = mols of vapor from plate to plate per unit time

D = mols of distillate withdrawn as product per unit time

x_d = mol fraction of the more volatile component in the distillate

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[1] McCabe and Thiele, Graphical Design of Fractionating Columns. Ind. Eng. Chem. *17*, 960 (1925). Walker, Lewis, McAdams, and Gilliland, ibid., page 545. Badger and McCabe, ibid., page 336. Hausbrand, ibid., page 99. Carey, Chemical Engineers Handbook, ibid., page 1176.

[2] Underwood, The Theory and Practice of Testing Stills. Trans. of the Institution of Chemical Engineers. *10,* p. 112 (1932).

[3] Brown, Distillation. Trans. Am. Inst. Ch. E., *32*, 321 (1936).

[4] Loc. cit.

By a material balance it can be shown that

$$V = R + D \tag{11}$$

In the McCabe and Thiele method, as well as in other methods, the plates are assumed to act as ideal plates and the composition of the vapor, y_n^*, from any plate "n" is assumed to be in equilibrium with the composition of the liquid, x_n, on the plate, the relationship being determined from a vapor-liquid equilibrium diagram for the particular binary mixture under consideration. The actual composition of the vapor, however, is usually weaker in the more volatile component than is indicated by the vapor liquid equilibrium phase relationship. Murphree[1] developed an equation which corrects for this departure from equilibrium values of the vapor. This correction factor is known as the Murphree Efficiency factor. The equation is

$$E_m = \frac{(y_n - y_{n-1})}{(y_n^* - y_{n-1})} 100 \tag{12}$$

where E_m = Murphree plate efficiency factor
y_n = actual composition of the more volatile component in the vapor leaving plate n (mol fraction)
y_{n-1} = actual composition of the more volatile component in the vapor leaving plate "$n - 1$" and entering plate "n" (mol fraction)
y_n^* = composition of the more volatile component in the vapor in equilibrium (from vapor liquid equilibrium diagram) with the liquid of composition, x_n, on plate n (mol fraction)

Carey[2] gives detailed calculations concerning the Murphree efficiency factor. Carey, Griswold, Lewis, and McAdams[3] reported data on an experimental bubble cap column. Peavy and Baker[4] presented data on the distillation of ethyl alcohol and water and showed that plate efficiencies as high as 120% resulted from concentration gradients across the plate.

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[1] Murphree, Rectifying Column Calculations. Ind. Eng. Chem. 17, 747 (1925). Murphree, Graphical Rectifying Column Calculations. Ind. Eng. Chem. 17, 960 (1925).

[2] Carey, Chemical Engineers Handbook, ibid., page 1185.

[3] Carey, Griswold, Lewis, and McAdams. Plate Efficiency in Rectification of Binary Mixtures. Trans. Amer. Inst. Chem. Eng. 30, 504-19 (1933).

[4] Peavy and Baker. The Efficiency and Capacity of a Bubble Plate Fractionating Column. Trans. Amer. Inst. Chem. Eng. *33*, No. 3, 315-345 (1937).

LIVE STEAM DISTILLATION

Live steam distillation finds application in meeting the engineering requirement of the separation of volatile organic liquids from relatively non-volatile materials at temperatures below the boiling point of the distillate or any avoidable decomposition point of the liquid mixture; distillation of dewaxed lubricating fractions in petroleum refining; separation of volatile liquids from absorption oils as in the debenzolization of light oil; and many others.

The reduction in temperature in live steam distillation is based upon the principle that, in a mixture of immiscible liquids each component liquid exists in its own phase and exerts its normal equilibrium vapor pressure. The total vapor pressure exerted by a mixture of immiscible liquids is the sum of the vapor pressure of the individual components at that temperature; therefore, the temperature of steam distillation at atmospheric pressure must always be less than 100° C. or 212° F.

The composition of the vapors evolved may be computed by an equation[1] relating the weights of the two components in the distillate with their respective molecular weights and vapor pressure at the temperature of distillation.

$$\frac{W_a}{W_b} = \frac{P_a M_a}{P_b M_b} \tag{13}$$

where W_a and W_b = Weights of components "*a*" (usually water) and "*b*" respectively in the distillate, in pounds

P_a and P_b = Vapor pressure of components "*a*" and "*b*" respectively at the temperature of distillation

M_a and M_b = Molecular weight of components "*a*" and "*b*" respectively

Robinson and Gilliland[2] shows the advantages of the use of vacuum steam distillation in the debenzolization of light oil.

EXPERIMENT NO. 25

Determination of Liquid-Vapor Equilibria

Object. 1. To determine vapor-liquid equilibria relationships of a binary mixture at the boiling point, and compare results obtained with recorded values from the literature.

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[1] Walker, Lewis, McAdams, and Gilliland, ibid., page 537. Badger and McCabe, ibid., page 368. Carey, Chemical Engineers Handbook, ibid., page 1163.

[2] Ibid., pages 148-154.

2. To compare observed experimental results with values computed by Raoult's Law.

Equipment. The apparatus recommended for the determination of vapor-liquid equilibria relationships is the one used by Carey and Lewis [1] as modified by Meyer.[2] The apparatus is illustrated in Fig. 5, as constructed in our Chemical Engineering Laboratories, Ohio State University.

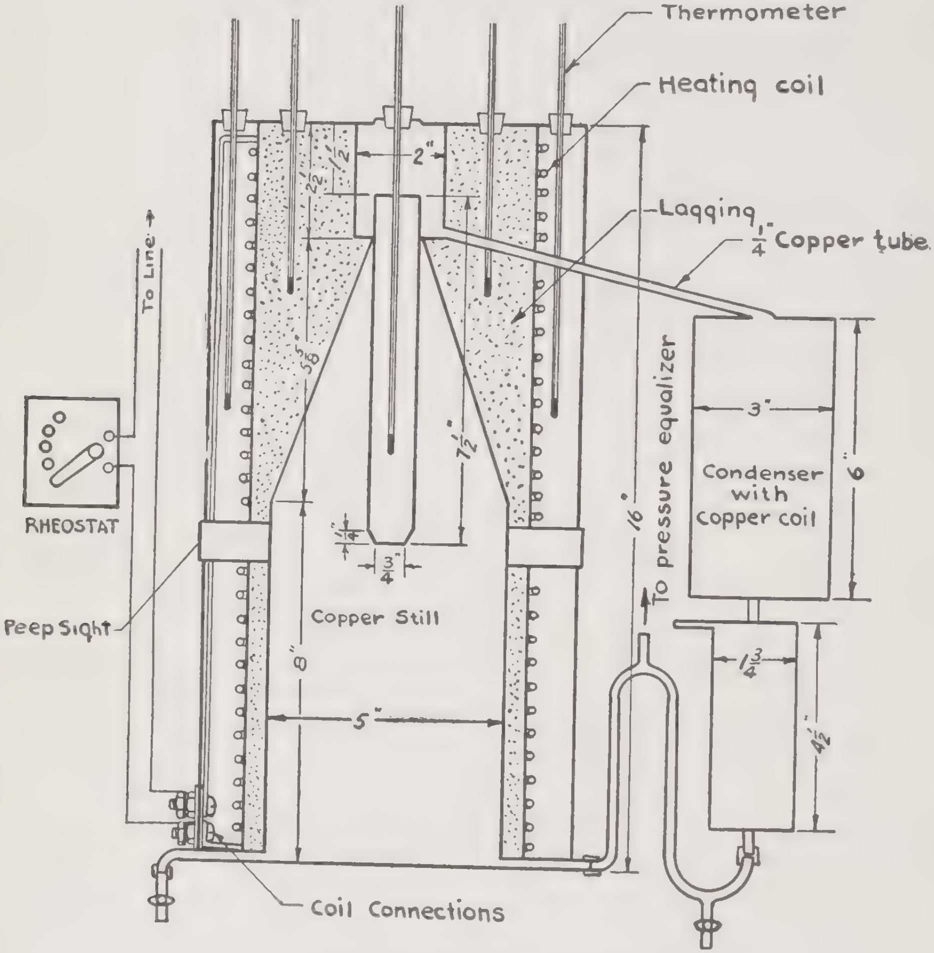

FIG. 5.—Equilibrium Still.

Procedure. Make up a series of samples of a binary mixture (such as, benzol-toluol; acetone-water; methanol-water; methanol-acetone; benzol-carbontetrachloride) for which equilibria data are to be determined.

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[1] Loc. cit.

[2] Meyer, Distillation Equilibrium Diagrams. Chemical Engineering Thesis, The Ohio State University, 1936.

Check compositions from measurements of specific gravity (using pycnometers, hydrometers, or Westphal Balance) or refractive index (using Abbé Refractometer).

Place in the still approximately 400 cc. of the binary mixture of the desired concentration. Record barometric pressure.

Circulate cooling water through the condenser jacket, apply heat gently at the bottom of the still by means of a bunsen burner, and turn on electric heating coil in still jacket. Carry out distillation at a rate of about 10 cc. per minute.

Adjust the electric heating coil rheostat so that the temperature of the insulating material is the same as the temperature of the vapor in the still.

Continue run for at least one hour, returning the condensed vapors to the still, after the temperature of the vapor remains constant and refluxing of liquid back to the still has ceased, as observed by watching the tip of the vapor tube through the peep sight. This will insure that contents of receiver will be changed at least ten times and that a steady state of equilibrium is being maintained.

At the end of the run take samples of condensate from condensate receiver and of still liquor in 250 cc. Erlenmeyer flasks immersed in ice baths. Cork samples immediately.

Determine composition of samples from measurements of specific gravity or refractive indices.

Determine boiling point of sample of still liquid using Cottrell,[1] Boiling Point Apparatus.

Preliminary Report: Before writing preliminary report and obtaining approval to proceed with the experimental program, examine and study the apparatus, the instructions given above, the requirements for the final report, and the pertinent references.

The preliminary report should contain the following:

Detailed experimental procedure, points of sampling, methods of control, and blank data sheets tabulating observations to be taken.

Detailed discussion of the following:

a. Sources of possible error such as: flash vaporization of condensed vapors returning to the still, sampling, and analytical measurements. Suggest the methods which seem best to avoid the errors enumerated.

b. The effect of superheating the vapors on equilibrium conditions.

c. State why the properties of most solutions deviate from the laws of ideal solutions.

d. Illustrate by use of phase diagrams how binary mixtures may be classified.

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[1] Cottrell, On the Determination of Boiling Points of Solution. J. Am. Chem. Soc. *41*, 721 (1919).

REPORT

Detailed report, besides the usual clarifying organization, should contain the following:

1. All observed and calculated data.
2. Chart containing curves of composition of liquid versus composition of vapor from the following sources:
    - (a) Values experimentally determined.
    - (b) Recorded values for identical liquid composition.
    - (c) Similarly, values computed by Raoult's Law.
3. Discussion of sources of error in the experiment.
4. Under recommendations bring out any suggested modifications in equipment, technique, and methods of calculations with reasons for same.

SAMPLE DATA AND CALCULATIONS

Run No. 1	Experimental Values	Recorded Values *	Assumed and Calc. Values
Weight per cent acetone in water in still (liquid)	66.0	66.0	66.0
Mol fraction acetone in water in still (liquid)	0.375	0.375	0.375
Corresponding boiling temp. Mixture, ° C.	60.6	61.0	78.0
Weight per cent (from sp. gr. tables) acetone in water (vapor) in equilibrium with the composition of the liquid in still	93.8	94.36	88.4
Computed mol fraction acetone in water (vapor) in equilibrium with the composition of liquid in still	0.825	0.841	0.78

* *Principles and Practise of Industrial Distillation.* E. Hausbrand. Translated from the Fourth German Edition by E. Howard Tripp. John Wiley & Sons, New York City, 1928. Page 215.

Run No. 1

1. Conversion Weight Per Cent to Mol Fraction:

$$\text{Mol Fraction Acetone} = \frac{\dfrac{\text{Wt. \% Acetone}}{\text{Mol Wt. Acetone}}}{\dfrac{\text{Wt. \% Acetone}}{\text{Mol Wt. Acetone}} + \dfrac{\text{Wt. \% Water}}{\text{Mol Wt. Water}}}$$

$$= \frac{\dfrac{66}{58.06}}{\dfrac{66}{58.06} + \dfrac{(100 - 66)}{18}} = 0.375$$

2: Calculation Boiling Temperature of Mixture (Raoult's Law):

Assuming Raoult's Law to apply for a given liquid mixture, a plot of vapor pressures of any component in the liquid mixture at a constant temperature versus its mol fraction in the mixture must give a straight line. The total pressure of the solution which is the sum of the partial vapor pressures of its components must also be a straight line.

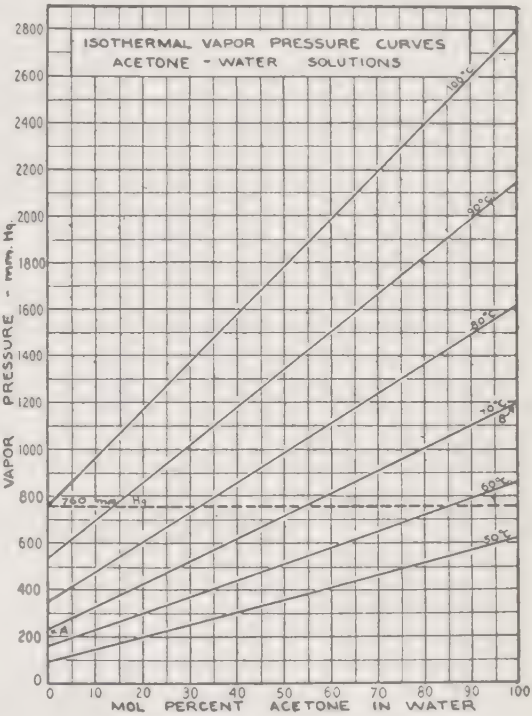

FIG. 6.—Isothermal-vapor Pressure Curves. Acetone-Water Solutions.

For example in Fig. 6 the straight line *AB* represents the total vapor pressure of all possible mixtures of acetone in water at 70° C. It was constructed from two points: *A*, which represents the vapor pressure of pure water at 70° C.[1], and *B*, which represents the vapor pressure of pure acetone at the same temperature.[2] The curves for the other temperatures were plotted in like manner.[3]

The intersection of the line *AB* with the 760 mm. isobar represents the composition of acetone in water (0.55 mol fraction) that will boil at a temperature of 70° C. and under atmospheric pressure. By interpolation between the intersection of lines *AB* and *CD* with the 760 mm. isobar it was found that a 0.375 mol fraction acetone-water mixture would boil at 78° C. if Raoult's Law applied.

3: Calculation Mol Fraction in Vapor in Equilibrium with Liquid:

Assuming that Raoult's Law and the Laws of Dalton and Amagat[4] apply, the composition of the vapor (y_a) in equilibrium with the composition of the liquid ($x_a = 0.375$ mol fraction acetone) at a temperature of 78° C. and a total pressure of 760 mm. Hg, was calculated as indicated below:

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[1] Joseph H. Keenan, Steam Tables and Mollier Diagram. The American Society of Mechanical Engineers. New York City, 1930.

[2] Chemical Engineers Handbook, pp. 326-335.

[3] If vapor pressure data were not available at a series of temperatures they could be determined from a Duhring Plot or a Cox Chart. O. A. Hougen and K. M. Watson. Industrial Chemical Calculations. John Wiley & Sons, New York City, 2nd Edition 1936, pp. 72 and 75.

[4] Hougen and Watson, ibid., page 47.

If P_a = the vapor pressure of pure acetone at 78° C., obtained from the Duhring line of acetone as given by Hougen and Watson[1] = 1580 mm., then $y_a = P_a x_a = \dfrac{1580 \times 0.375}{760} = 0.78$ mol fraction acetone.

EXPERIMENT NO. 26

SIMPLE DISTILLATION OF A BINARY MIXTURE

Object. 1. To compare observed experimental results obtained in simple distillation of a binary mixture with values computed by the Lord Rayleigh equation.

2. To determine the performance characteristics of a simple batch still by plotting a graphical log of experimental data, and calculating heat and material balances.

Equipment. A type of still which may be used for this problem is shown in Fig. 4. The column and reflux condenser are cut out, by closing appropriate valves, allowing the vapors to pass directly from the still kettle to the final condenser where they are condensed and cooled. The condensate may be collected in the calibrated receivers "R_1" and "R_2," or tared containers placed on scales.

The various temperature measurements may be taken by resistance thermometers connected to a central instrument board as illustrated in Fig. 4, or by any other common type of temperature measuring instrument, as: thermocouples, or mercury thermometers.

The quantity of cooling water to the condenser may be measured by water meters as illustrated, or by calibrated orifices, or by weigh tanks.

Rate of distillation may be measured by any one of the following methods: rotameter, calibrated weir, sight glass observation, calibrated or weigh tanks. Measurement by rotameter is recommended, as it gives instantaneous readings, and it requires little or no attention.

Steam quality may be determined by the use of either a throttling or separating calorimeter.

The steam consumption may be determined by discharging the condensate through a steam trap into tared receivers placed on platform scales.

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[1] Loc. cit.

Steam coil, still, and condenser pressures may be determined by mercury manometers.

Composition of mixtures may be determined by refractive indices or specific gravity measurements, and have to be taken with extreme care.

Graphical integration underneath the curve of x versus $l/y - x$ may be determined by means of a polar planimeter, counting the squares underneath the curve, Simpson's Rule or by one of the common graphical integration methods.[1]

Procedure. Set the necessary valves so that still may be operated as a simple batch still.

Check carefully.

Make up a binary mixture in the still tank, such as, acetone and water; benzol and toluol; methanol and water, weight and composition of charge as specified by suitable instructions.

Precautions. In working with organic liquids, remember that they are highly inflammable; no flames, smoking, or electrical sparking equipment should ever be tolerated in the vicinity of the still. Beware of *toxicity.* Beware of air-vapor mixtures. They are *highly dangerous* as *explosive hazards. Every part of* equipment must be *electrically grounded.*

Never start anything you cannot stop; obviously, this is an excellent rule from the standpoint of safety precaution.

Provide adequate receiving vessels. Ascertain that the equipment is properly vented. Allow a substantial amount of water to flow through the final condenser, adjust to maintain desired temperature of distillate after distillation begins.

Heat still charge, regulating the steam valve so that the temperature rise of the liquid in the still is about 5° C. per minute. Beware of inattention to rate of heating. Dangerous or galloping flood flow may result. When the vapor line above the still becomes warm, shut off the steam valve completely and watch the rise in temperature. If very slow, then only, crack the steam valve and open gradually at intervals. When distillation begins again shut off steam entirely and at once crack the steam valve and slowly open if the distillate stream definitely slows down. Careful attention must be given the heating until the rotameter reading of the distillate indicates the rate of vaporization desired or specified by the instructions.

Change distillate receivers when full or when the composition of the liquid in the still corresponds to that specified for the various runs.

Discontinue distillation when the temperature of the liquid in the still has reached the boiling point of the less volatile component.

Determine residue in the still by weighing, cooling first to a temperature at which it may be safely handled.

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[1] Running. Trans. Amer. Inst. Chem. Eng. Vol. XXIII, page 159 (1929).

PRELIMINARY REPORT

By examination and study of the still indicate on a sketch the valves to be opened and closed, points of sampling, and methods of control.

Submit, after squad conference under supervision of the foreman, data sheets for the observations to be made, one for each station or observer.

Set up a detailed material and heat balance, indicating the data to be taken, assumptions, and calculations that must be made. Indicate alternative methods of checking heat and material balances.

Indicate by sketch an Enthalpy Concentration Diagram,[1] and show how such a diagram (Merkel Chart) could be used for heat balance calculations. Indicate how such a diagram [2] may be constructed from data obtained from the literature. Show why such refinement in calculations is or is not [3] necessary for the particular binary used. Give the equations for approximate calculations of specific heats, and latent heat of vaporization of solutions.

Discuss the effect of refluxing of condensed vapors to the still on (a) composition of the distillate, (b) amount of distillate obtained as compared to calculated amount by Lord Rayleigh equation.

Report to instructor for a quiz on this problem. Obtain his approval for proceeding with the experiment.

REPORT

Detailed final report should contain the following:

1. Objectives.
2. Observed and calculated data.
3. Comparison of observed data of composition and amount of distillate with values computed by the Lord Rayleigh equation.
4. A plot of per cent vaporization versus cumulative composition of the distillate, observed and calculated.
5. Graphical log or plot of all observed and calculated data with detailed discussion of relationships and discrepancies between calculated and observed results, as: composition and weight of distillate, and temperature of liquid in the still.
6. Complete material balance.
7. Complete heat balance indicating clearly all assumptions made and their justification.

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[1] McCabe, The Enthalpy Concentration Chart. Trans. Amer. Inst. Chem. Eng. 31, 129 (1934).

[2] Hougen and Watson, ibid., page 357.

[3] Elliott, Distillation Principles. Ernest Benn Ltd., 1925. Page 146 et seq.

SAMPLE DATA AND CALCULATIONS

Material Balance

	Experimental		From Tables and Calculations			
	Total Pounds	Sp. Gr. 20° C.	% by Wt. Acetone	% by Wt. Water	Pounds Acetone	Pounds Water
A. Input:						
Charge to still	350.0	0.958	29.65	70.35	103.8	246.2
B. Output:						
Residue in still	224.0	0.997	1.00	99.00	2.2	221.8
Distillate	123.0	0.847	81.00	19.00	99.6	23.4
Total	347.0	......	....	....	101.8	245.2
C. Unaccounted for	3.0	......	....	....	2.0	1.0

Heat Balance Basis 32° F.

	Pounds	Enthalpy B.t.u./Lb. Above 32° F.	Total Heat B.t.u.'s	% of Total
A. Input:				
a. Charge in still	350	34.4	12,040	2.2
b. Steam to still	126	1122.0	141,370	25.9
c. Condenser cooling water	7140	55.0	392,100	71.9
Total	....	....	545,510	100.0
B. Output:				
a. Residue in still	224	180.0	40,320	7.1
b. Steam condensate	126	143.0	18,010	2.8
c. Condenser cooling water	7140	64.0	457,000	83.3
d. Distillate	123	35.3	4,330	0.8
e. Total accounted for	....	....	519,660	95.0
C. Radiation and unaccounted for	....	....	25,850	4.7

Observed and Computed Weight and Composition of Distillate

Run Number	1	2	3	4
a. Charge to still. Pounds	350	350	350	350
b. Comp. charge % by wt. acetone	29.65	29.65	29.65	29.65
c. Composition liquid in still at end of run, % acetone by wt	24.24	16.06	5.50	1.00
d. Weight of distillate calculated by Lord Rayleigh equation. Pounds	30.0	67.0	106.5	118.0
e. Cumulative weight of distillate. Observed. Pounds	28.0	64.5	102.0	123.0
f. Composition of distillate calculated by Lord Rayleigh Equation, % acetone by wt	87.3	86.1	84.7	83.3
g. Composition distillate. Observed % by wt. acetone	92.0	90.7	88.3	81.0
h. % liquid in still vaporized, based on charge	8.0	18.3	29.1	35.0

Heat Balance Calculations. *Total Heat Content.* The total heat content of the liquid entering into each item of the heat balance may be calculated by the following equation:

$$H_l = h_l W_l \tag{14}$$

Where (Reference temperature 32° F.)

H_l = Total relative heat content of the liquid, B.t.u. above 32° F.
h_l = Relative enthalpy of the liquid, B.t.u. per pound above 32° F.
W_l = Weight of liquid in pounds.

Condenser Cooling Water. In the case of condenser cooling water the values of the enthalpy of water at temperature in question may be obtained from steam tables.[1]

For example, the average temperature of inlet cooling water was 87° F. Enthalpy of water at 87° F. was found to be 55 B.t.u. per pound above 32° F. Therefore,

$H_l = 55 \times 7140 = 392{,}100$ B.t.u., total relative heat content of inlet cooling water.

Charge to Still – Distillate – Still Residue. Since these items involve mixtures, the heat of solution effects may have a profound influence upon the resulting enthalpy. If enthalpy diagrams (Merkel Charts)[2] are available for this particular system they should be used in these calculations. A less rigorous alternative method, sometimes resorted to, is that given by Creagh and the authors.[3] The enthalpy, however, may be merely approximated from the specific heats of the components.[4] The equation of the enthalpy of a mixture is then:

$$h_l = (c_a w_a + c_b w_b)(t - 32) \tag{15}$$

Where:

c_a, c_b = specific heats of components a and b in the mixture.

w_a, w_b = weight fractions of a and b in the mixture.

t = temperature of mixture.

---

[1] Keenan and Kayes, Steam Tables and Mollier Diagrams. The American Society of Mechanical Engineers. 2nd Ed. 1936.

[2] McCabe, loc cit. Hougen and Watson, ibid., page 357.

[3] Creagh, Koffolt, and Withrow. Thermodynamic Investigation of the Evaporation of Concentrated Potassium Sulphate Liquor from Polyhalite. Trans. Amer. Inst. Chem. Eng. 32, 554 (1936).

[4] C. Elliott, loc. cit.

In the example cited the total relative heat content of liquid charged to the still was calculated thus:

$$\begin{aligned} h_l &= (0.528 \times 0.2965 + 1.00 \times (1 - 0.2965)\ (72 - 32) \\ &= (0.1563 + 0.7035)\ 40 \\ &= 34.4 \text{ B.t.u. per pound} \\ H_l &= 34.4 \times 350 = 12{,}040 \end{aligned}$$

Weight of Distillate. *Calculated by Lord Rayleigh Equation.* Substituting observed values of W_0, x_0, and x_r of Run No. 3 in the Lord Rayleigh equation

$$2.3 \log \frac{350}{W_r} = \int_{5.5}^{29.65} \frac{dx}{y - x} \tag{1}$$

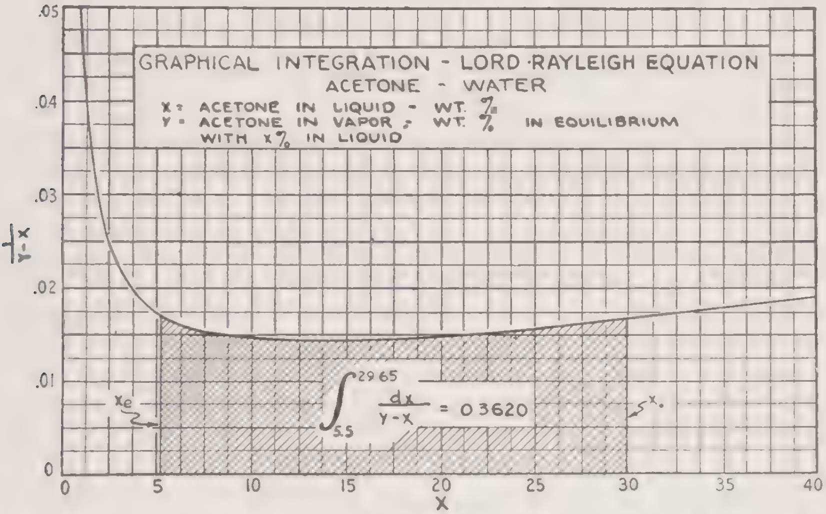

FIG. 7.—Graphical Integration of Lord Rayleigh Equation. Acetone-Water.

Value of integral $\frac{dx}{y - x}$ was determined by graphical integration by measuring shaded portion of the curve (Fig. 7) between the limits $x_0 = 29.65$, and $x_r = 5.5$.

Area as determined by planimeter = 7.24 square inches.

1 square inch = 5 × 0.01 = 0.05 integration units.

Value of the integral is therefore 7.24 × 0.05 = 0.3620 integration units.

$$\begin{aligned} 2.3\ (\log 350 - \log W_r) &= 0.3620 \\ W_r &= 243.5 \text{ pounds} \end{aligned}$$

$$\text{Weight of distillate (calc.)} = W_0 - W_r = 350 - 243.5 = 106.5$$

Composition of Distillate. *Calculated by Lord Rayleigh Equation.*

$$x_d = \frac{(W_0x_0 - W_rx_r) \times 100}{(W_0 - W_r)} = \frac{(350 \times 0.2965 - 243.5 \times 0.0552) \times 100}{350 - 243.5}$$

$$= 84.7\%$$

$$\% \text{ vaporized} = \frac{103 \times 100}{350} \quad 29.1\%$$

EXPERIMENT NO. 27

Simple Continuous Distillation

Object. 1. To determine the effect of varying rate of vaporization on composition of distillate and residual liquid in simple continuous distillation of a binary mixture.

2. To compare experimentally determined compositions and weights of distillate with those calculated from vapor-liquid equilibria data in simple continuous distillation of a binary mixture.

3. To compare by calculation the percentage of a given mixture vaporized at a given temperature by simple continuous distillation, with that obtained by simple batch distillation to the same final temperature.

Equipment. A type of still suitable for this problem is the same as for Experiment No. 26, "Simple Distillation of a Binary Mixture," and as illustrated in Fig. 4. However, additional auxiliary equipment and measuring devices must be installed, as outlined below, to meet the requirements of continuity of operation.

Calibrated closed storage and feed tanks can be constructed from fifty gallon drums vented to the atmosphere, equipped with: air, steam, or water driven agitators; liquid level gauge glasses, and openings for making up, butting up, and draining of tanks.

Feed solution to the still may be controlled and measured by a rotameter, calibrated closed weir tank, or sharp edge orifice and checked by time-volume measurements of calibrated feed tanks.

Rate and quantity of residual liquid withdrawn from the still may be measured by rotameter, weir, or weigh tanks, and the checking of constancy of level in the still by gauge glass attached thereto.

Procedure. Make up binary mixtures in feed and storage tanks as specified by the instructor, and in the still kettle corresponding to that calculated for the particular run in the preliminary program report. In working with *organic liquids*, observe the *precautions emphasized* in Experiment 26 in regard to inflammability and air-vapor explosion hazards.

Allow ample water to flow through the condenser. Adjust to maintain desired temperature of distillate after distillation begins.

Heat still charge, regulating the steam valve as in previous runs so that the temperature rise of the liquid in the still is about 5° C. per minute. When the vapor line above the still becomes hot, shut off steam valve completely.

Open the feed valve so that the rate of feed as indicated by the instrument used corresponds to the rate specified by the instructor. Then crack the steam valve and open gradually, employing previously used precautions, till the rate of vaporization corresponds to that calculated for the particular run in the preliminary program report.

Crack the residual liquid valve so a constant level is maintained in the still kettle.

Continue the run at least thirty minutes after the still has attained a steady operating state as indicated by the constancy of the rate of feed, temperature of liquid in the still, composition of feed, liquid in the still, and distillate, constant level in the still, rate of vaporization, rate of flow of residual liquid from the still, and running material balances of observed data.

By increasing the rate of vaporization and adjusting residual liquid valve make other runs. At end of the runs shut off feed and residual liquid valves and operate as simple batch still until the still liquid contains practically none of the more volatile liquid. At least four runs should be made varying the per cent of vaporization of feed approximately as follows: 20%, 40%, 60%, 80%.

Be prepared, if necessary, to make up more feed solutions during the experiment in storage tanks using distillate and *cooled* residual liquid from earlier runs. Use nomographic chart for calculating the amount of "make-up" and "butt-up" solutions required.

PRELIMINARY REPORT

By examination and study of the still indicate on a sketch the valves to be opened or closed, points of sampling, and methods of control.

Submit after squad conference, under supervision of the foreman, data sheets for the observations to be made, one for each station or observer.

Based upon the operating conditions assigned your group, as: composition of feed, and rate of feed vaporization, calculate the following: (a) temperature of distillation, (b) rate of withdrawal of residual liquid, and (c) composition of distillate and residual liquid.

Construct a nomographic chart [1] for the "make-up" and "butt-up" of feed solution using the following: pure materials, and liquids varying from minimum composition of residual liquid and maximum composition of distillate as calculated above, and in terms of the more volatile component.

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[1] Koffolt and Withrow, Graphic Control of Plant Solutions. Chem. and Met. Eng 46, 161 (1939).

Compare, by calculations, the percentage of a given mixture vaporized at a given temperature by simple continuous distillation with that obtained by a simple batch distillation to the same temperature. Calculate also the composition of distillate in each case.

Report to instructor for quiz on this problem. Obtain his approval to proceed with the experiment.

FINAL REPORT

Detailed final report should contain the following:

1. Objectives.
2. Graphical log of all observed and calculated data.
3. Chart comparing the experimentally determined compositions and weights of distillate with those calculated from vapor-liquid equilibria data in simple continuous distillation of binary mixture used in test runs.
4. A plot of per cent vaporization versus composition of the distillate, based on observed and calculated results.
5. Under recommendations bring out desired modifications in equipment. technique, and methods of calculations with reasons for change.

SAMPLE DATA AND CALCULATIONS

	Run No. 2
Composition of feed to still. Per cent acetone by weight	30.3
Average rate of feed (by rotameter). Pounds per minutes	2.8
Time of run. Minutes	40
Level of liquid in still. Start of run. Cm.	37.5
Level of liquid in still. End of run. Cm.	37.5
Quantity of feed. From feed tank measurements. Pounds	105
Liquid withdrawn from the still during the run. Pounds	85.5
Distillate collected. Pounds	19
Percentage feed vaporized	18.0
Composition of distillate. Experimental. Per cent acetone by weight	88.0
Composition of residual liquid. Experimental. Per cent acetone by weight	18.21
Composition of distillate calculated from vapor liquid equilibrium relationship, composition of feed, and ratio of weight of liquid withdrawn from still to distillate. Per cent acetone by weight	86.32
Composition of the liquid in equilibrium with composition of the distillate (86.32%)	17.5
Temperature of distillation. Observed Degrees C.	70.1
Temperature of distillation. From equilibrium diagram and graphically computed composition of liquid in still (17.5%). Degrees C.	72.5
Weight of distillate. Calculated from observed composition of still liquid and vapor liquid equilibria data. Pounds	18.5

Composition of distillate calculated from vapor-liquid equilibrium relationship, composition, and percentage of feed vaporized: This was computed from a plot (Fig. 8) of percentages of feed vaporized versus compositions of distillate (y), and noting from observed value of per cent vaporized the corresponding value of y. Where the percentage of feed vaporized, is as shown before, computed from the following equation:

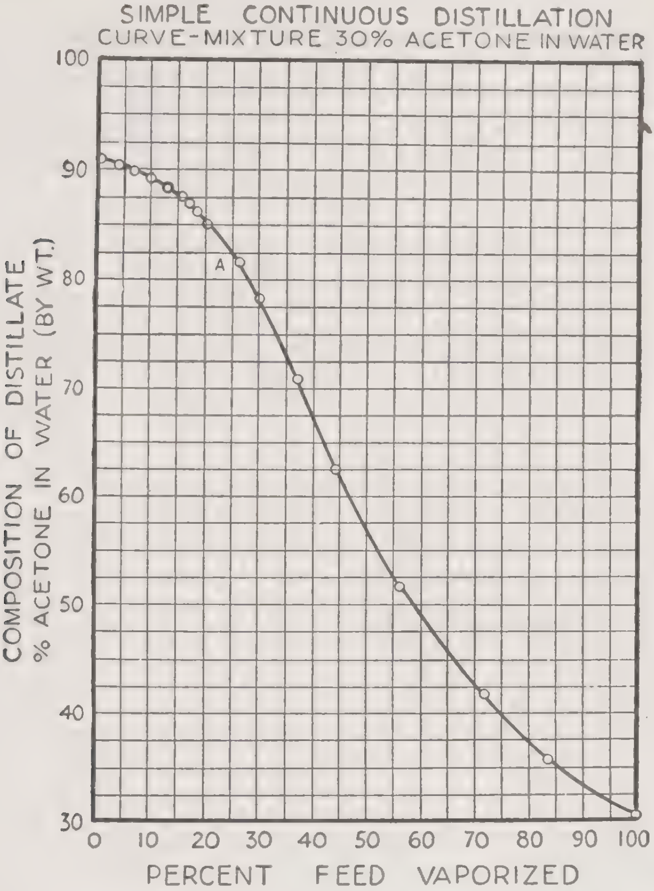

FIG. 8.—Simple Continuous Distillation Curve of a Mixture of 30.3 Percent Acetone in Water.

$$\% \text{ vaporized} = \frac{x_f - x_w}{y - x_w} \times 100 \tag{3}$$

For example, point A on the curve, Fig. 58, was obtained as follows: Let the composition of the distillate (y) = 81.93% by weight of acetone in water. From Fig. 1, the composition of the liquid (x_w) in equilibrium with y is 12.5% acetone in water. Since the composition of the feed is 30.3% acetone in water, then,

$$\% \text{ vaporized} = \frac{30.3 - 12.5}{81.93 - 12.5} \times 100 = 25.6$$

The other points on the curve were obtained in a corresponding manner.

Since 18% of the feed in the run illustrated under sample data was vaporized the composition of the distillate calculated under equilibrium conditions would be from Fig. 8 86.32% acetone in water.

Under these same conditions it can be seen from Fig. 1 that the composition of the residual liquid would be 17.5% acetone in water, and the temperature of distillation 72.5° C.

EXPERIMENT NO. 28

Vapor Enrichment by Partial Condensation (Dephlegmation)

Object. 1. To determine the performance characteristics of a partial condenser.

2. To compare experimentally determined compositions of distillate and condensate, in partial condensation of binary mixtures, with values computed for simple and differential partial condensers.

Equipment. The type of still that may be used for this problem is illustrated in Fig. 4. It will be noted that it is the same as used for the previous two problems, except that an additional condenser, and some means of measurement or calculation of the partial condensate must be provided. The flow of vapors and partial condensate formed in the partial condenser are parallel current and in the downward direction. Such an arrangement indicates that the partial condenser will behave more as a simple than a differential partial condenser, since the condensate formed is always in contact with the vapors throughout the condensing period. Counter current flow of vapors and condensate would have a tendency for the operation to approach differential rather than simple partial condensation.

The partial condensate formed in the partial condenser is best measured with a rotameter; it can, however, be measured by heat balance calculations. The distillate may be measured by the same methods.

By returning the distillate back to the still kettle as fast as it is condensed the performance characteristics of the partial condenser are more easily determined, as steady state conditions will thereby be obtained.

Operating Conditions. At least four runs are to be made varying the percentage of partial condensation of vapors from the still approximately as follows: 20%, 40%, 60%, and 80%.

If arrangement of the still is such that both parallel, and counter current flow in the partial condenser can be obtained, additional runs are suggested permitting thereby a comparison of the performance characteristics of these two types of condensers.

Procedure. Make up a binary mixture in the still kettle, such as: acetone and water; benzol and toluol; methanol and water. Weight and composition of the charge to be specified by the instructor. In working with organic mixtures always be on guard against explosive mixtures of these materials and air. Beware of *toxicity*. No flames, smoking, or electrical sparking equipment should ever be tolerated in the vicinity of the still. *Every part* of the equipment must be *electrically grounded.*

Allow a large amount of water to flow through the final condenser, adjust to maintain the desired temperature of distillate after distillation begins.

Allow a sufficient amount of water to flow through the partial condenser to completely condense all vapors that will be evolved from the still.

Set necessary valves so that the distillate line is open to the still kettle.

Heat up still charge so that the temperature rise of the liquid in the still is about 5° C. per minute. When the vapor line above the still becomes hot, shut off steam valve completely. Crack and open the steam valve gradually as suggested in the previous problems until the desired rate of vaporization is obtained as indicated by the rate of flow of partial condensate from the partial condenser to the still kettle.

Adjust water to the partial condenser so as to condense the percentage of total vapors from the still as specified for the particular run.

Continue the run for at least thirty minutes after steady state conditions have been attained, which will be indicated by constancy of the following observations: rate of flow of partial condensate and distillate from the respective condensers, temperature of the liquid in the still and vapors to and from the partial condenser, composition of still liquid, partial condensate, and distillate.

Make other runs by increasing the percentage of partial condensation of vapors from the still as suggested above.

At the end of the run allow distillate to flow into receivers instead of still kettle. Operate as simple batch still until the still liquid contains practically none of the more volatile liquid.

PRELIMINARY REPORT

Before writing preliminary report and obtaining approval to proceed with the experiment examine and study the still, the instructions given above, the requirements for the final report, and the pertinent references.

Preliminary report should contain the following:

1. A diagrammatic sketch of the set-up to be used indicating the valves to be opened or closed, points of sampling, and methods of control.

2. Data sheets of observations to be made, one for each station and observer. These to be prepared in squad conference under supervision of the squad foreman.

3. Based upon the operating conditions assigned to your group, as: (a) composition of the charge, (b) rate of vaporization, (c) percentage of vapors to be condensed in the partial condenser for the various runs, calculate the following: (a) temperature of distillation, (b) composition of the partial condensate and distillate under conditions of simple and differential partial condensation, and (c) temperature of the vapor from the partial condenser under conditions of simple and differential partial condensation.

REPORT

Detailed report besides the usual clarifying organization should contain the following:

1. Objectives.
2. All observed and calculated data.
3. Chart containing the following curves plotted against the percentage of the vapors condensed in the partial condenser.
    (a) Composition of the distillate (observed).
    (b) Composition of the distillate (calculated) assuming for each run both simple and differential partial condensation.
    (c) Composition of the partial condensate (observed and calculated) as in (b) above.

SAMPLE DATA AND CALCULATIONS

	Run No. 4
Charge to the still. Acetone-water mixture. Pounds (observed)	417
Composition of liquid in the still. Per cent acetone by weight (observed)	45.6
Weight of vapors condensed in partial condenser. Pounds per minute (observed)	3.85
Weight of distillate from final condenser. Pounds per minute (observed)	0.96
Weight vaporized in the still. Pounds per minute (calculated)	4.81
Percentage of vapors from the still condensed in partial condenser. Per cent	80.0
Composition of the vapors from the still. Per cent acetone by weight, calculated from material balance	92.58
Composition of the vapors from still. Per cent acetone by weight obtained from vapor liquid equilibrium diagram (Fig. 1)	93.04
Composition of distillate from final condenser. Per cent acetone by weight (observed)	95.26

Composition of partial condensate from partial condenser. Per cent acetone by weight (observed)	91.92
Composition of distillate. Per cent by weight (calculated), assuming simple partial condensation	96.8
Composition of distillate. Per cent acetone by weight (calculated), assuming differential partial condensation	99.75
Composition of partial condensate. Per cent acetone by weight (calculated), assuming simple partial condensation	92.1
Composition of partial condensate. Per cent acetone by weight (calculated), assuming differential partial condensation	90.6

Calculation of the Composition of Vapor from the Still:

Let V_1 = pounds of vapor of composition y_1 (weight per cent) per minute from the still to the partial condenser.

V_2 = pounds of residual vapor of composition y_2 (weight per cent) per minute from partial condenser to final condenser (distillate).

L_2 = pounds of partial condensate of composition x_2 (weight per cent) per minute from partial condenser to the still.

Then by material balance equations:

$$V_1 = V_2 + L_2 \tag{16}$$

$$V_1 = 0.96 + 3.85 = 4.81 \text{ pounds}$$

and

$$y_1 = \frac{V_2y_2 + L_2x_2}{V_1} \tag{17}$$

$$y_1 = \frac{0.96 \times 95.26 + 3.85 \times 91.92}{4.81} = 92.58\% \text{ by weight acetone}$$

Calculation of the Composition of Residual Vapor (Distillate), and the Partial Condensate Assuming *Simple Partial Condensation:*

The composition of the residual vapor may be obtained from the simple partial condensation curve A (vapor of 93.04% acetone by weight), Fig. 9, in which the per cent vapors condensed in the partial condenser are plotted against composition of the more volatile components in the residual vapor (distillate), and noting from the observed value for the particular run of the per cent vapors condensed, C, the corresponding value of y_2. For example, in Run No. 4 (Sample Data) 80.0% of the vapors were condensed in the partial condenser, which gave from curve A, Fig. 9, a composition of 96.8% for the residual vapor (distillate) assuming simple partial condensation.

The composition of the partial condensate, x_2, which is in equilibrium with the composition of the residual vapor, y_2 (96.8%), was obtained from the vapor liquid equilibrium diagram for acetone and water, Fig. 1, and was noted to be 92.0% acetone, and the final temperature of condensation 58° C.

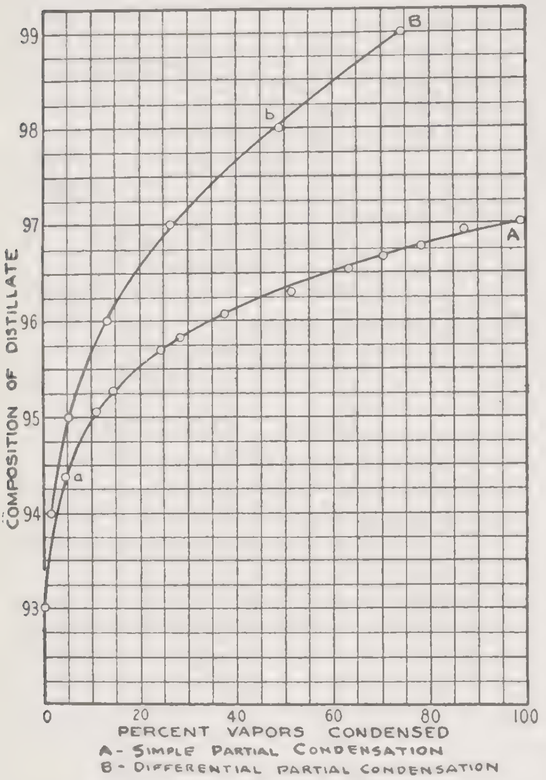

Fig. 9.—Simple and Differential Partial Condensation Curves of a Mixture of 93.04% Acetone in Water.

The points of curve *A*, Fig. 9, were constructed from calculated data. As an illustration, the calculation of one of the points, *a*, is given below.

Let the composition of the residual vapors from a simple dephlegmator be 94.36% acetone. From Fig. 1, the composition of the partial condensate, x_2, in equilibrium with this composition of the vapor (94.36%) is 66% acetone by weight. Since for this particular run the composition of the liquid (x_1) in the still was 45.6% acetone by weight the corresponding composition of the vapor, y_1, in equilibrium with this composition (45.6%) is, from Fig. 1, 93.04% acetone by weight. *C*, the per cent of vapors condensed in the partial condenser, may be computed by the principle of inverse proportions:

$$C = \frac{y_2 - y_1}{y_2 - x_2} \times 100 = \frac{(94.36 - 93.04)100}{(94.36 - 66)} = 4.66\% \qquad (6)$$

Therefore the coordinates for point "*a*" are: 4.66% (per cent vapors condensed), and 94.36%, composition of residual vapors (distillate).

The other points of curve *A* were obtained by a similar method of calculation.

Calculation of the Composition of Residual Vapor (Distillate), and Partial Condensate Assuming *Differential Partial Condensation:*

The composition of the residual vapor may be obtained from the differential partial condensation curve *B*, Fig. 9, in which the per cent vapors condensed in the partial condenser are plotted against the composition of the more volatile components in the residual vapor (distillate), and noting from the observed value for the particular run of the per cent vapors condensed (C), the corresponding value of y_2. For example, in Run No. 4 (Sample Data) 80.0% of the vapors were condensed in the partial condenser, which gave from curve B, Fig. 9, a composition of 99.75% acetone by weight for the residual vapor (distillate) assuming differential partial condensation.

The composition of the partial condensate, x_2, was obtained by a material balance calculation as follows:

$$x_2 = \frac{V_1 y_1 - V_2 y_2}{L_2} \tag{17}$$

$$x_2 = \frac{4.81 \times 92.58 - 0.96 \times 99.75}{3.85} = 90.6\%$$

The points on curve B, Fig. 9, were constructed from calculated data. As an illustration, the calculation of one of the points, b, is given.

Let the composition of the residual vapors from a differential partial condenser be 98% acetone by weight. The weight of vapor from the still to the partial condenser (V_1) be 100 pounds. Since for this particular run the composition of the liquid, x_1, in the still was 45.6% acetone by weight the corresponding composition of the vapor, y_1, in equilibrium with this composition (45.6%) is, from Fig. 1, 93.04% by weight of acetone.

The pounds of residual vapor can be computed by substituting the above values in the previously mentioned differential partial condenser Equation (9)

$$2.3 \log \frac{100}{V_2} = \int_{93.04}^{98} \frac{dy}{y - x} \tag{9}$$

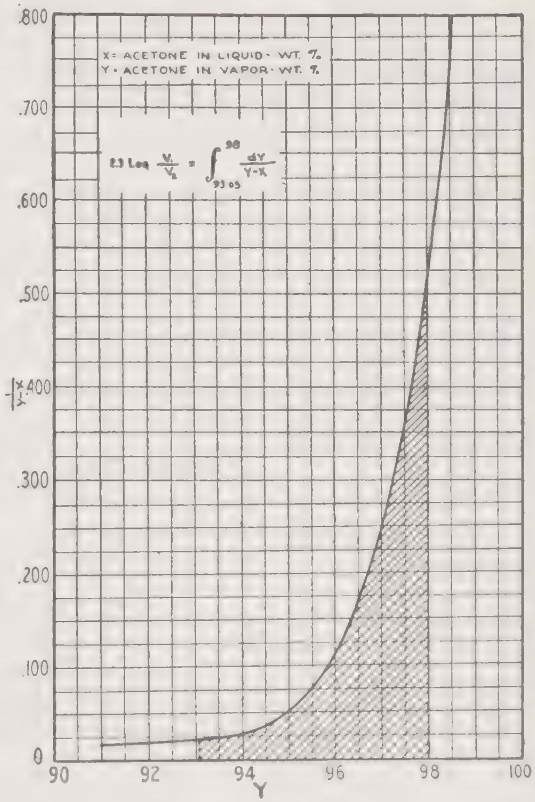

Fig. 10.—Graphical Integration Chart. Differential Partial Condensation. Acetone and Water.

The value of the integral $\int_{93.04}^{98} \frac{dy}{y - x}$ was determined by graphical integration by measuring the shaded portion of the curve (Fig. 10) between the limits $y_1 = 93.04\%$ and $y_2 = 98\%$.

Area as determined by planimeter = 3.266 square inches.
1 square inch = 2 × 0.1 = 0.20 integration units.
Value of the integral = 3.266 × 0.20 = 0.6532 integration units.

$$2.3\,(\log 100 - \log V_2) = 0.6532$$
$$V_2 = 52 \text{ pounds}$$
$$\text{Pounds condensed in partial condenser} = 100 - 52 = 48$$

$$\text{Per cent of vapors condensed} = C = \frac{48 \times 100}{100} = 48\%$$

Therefore the coordinates of the point "*b*" are: 48% (per cent vapors condensed), and 98.00% (composition of residual vapors [distillate]).

The other points for curve *B* were obtained in a similar manner.

EXPERIMENT NO. 29

BATCH COLUMN STILL

Object. 1. To determine the performance characteristics of a batch column still.

2. To determine the Murphree plate efficiency of a batch column still under actual operating conditions, and total reflux.

3. To determine the reflux ratio required to produce a given rectification in a batch column still, and to compare these values with calculated minimum reflux ratio.

Equipment. The type of still that may be used for this problem is illustrated in Fig. 4. The vapor line from the still kettle to the reflux condenser is cut out by closing the appropriate valves. It is often recommended from the standpoint of ease of operation that only one condenser be used, as close and accurate control of the desired reflux ratio can be obtained (especially with unskilled operators) by regulating the valve in the line from the condenser to the top plate of the column and the distillate line.

The auxiliary equipment used for measuring temperatures, rate of reflux and distillate flow, quantity of steam condensate, water rate, and pressures is the same as discussed in the previous problems.

Returning the distillate to the still kettle as fast as it is condensed permits the attaining of steady state conditions and hence the obtaining of more accurate data for design and operating problems that follow.

Procedure. Set the appropriate valves so that the still may be operated as a batch column still. However, in order to insure steady state conditions, open necessary valves so that the distillate will flow back to the still kettle as fast as it is condensed. Open vent lines from distillate line and receivers to the atmosphere.

Check carefully.

Make up a binary mixture in the still kettle, such as: acetone and water; benzol and toluol; methanol and water. Record weight and composition of the charge as specified by suitable instructions. In working with organic liquids remember that they are highly inflammable; no flames, smoking, or electrical sparking equipment should ever be tolerated in the vicinity of the still. Beware of *toxicity*. Beware of air-vapor mixtures. They are *highly dangerous* as explosion hazards. Every part of the equipment must be electrically grounded.

Provide adequate receiving vessels. Allow a substantial amount of water to flow through the combination condenser and adjust to maintain the desired temperature after distillation begins. Distillate should never be warm to touch.

Heat the still charge, regulating the steam valve so that the temperature rise of the liquid in the still is about 5° C. per minute. Beware of inattention to rate of heating. When the vapor line above the still becomes warm, shut off steam valve completely and watch the rise in temperature. If very slow, then slowly crack the steam valve and open gradually at intervals so that the pressure in the still never exceeds 2 cm. mercury. Observe reflux and distillate sight glasses at all times. When the liquid appears in the reflux or distillate sight glasses, again shut off the steam entirely. By observation of gauge glasses attached to the various plates on the column determine whether the column is liquid plugged. Allow sufficient time for the liquid on the plates to drain back to the still kettle.

Close valve to distillate line so that the still will operate for the first run under conditions of total reflux. Check carefully to make sure that the system is properly vented to the atmosphere.

Crack the steam valve and slowly open so the rate of total reflux as indicated by the rotameter is as desired or specified by instructions.

As the still is being operated under conditions of total reflux and hence no product is withdrawn from the system constant state conditions will be attained, as denoted by the constancy of temperature readings of the liquid in the still and on the plates, rate of flow of reflux, and steam consumption. When these conditions are assured, samples should be taken of the still liquid, liquid on all the plates, and the reflux. Determine the composition of these samples using specific gravity hydrometers, Westphal Balance, pycnometer, or by refractive index measurements.

Start the second run by opening and adjusting the valve to the distillate line so that distillate is obtained. Adjust this valve and the valve in the reflux condensate line to the top plate so that the desired composition of product as specified by instructions is obtained.

As this distillate is being fed continuously into the still kettle a new set of constant state conditions will be attained, as denoted by the constancy of the temperature readings, reflux ratio, and steam consumption. When these conditions are assured, samples should be taken of the still liquid, liquid on all the plates, reflux, and distillate. Determine the compositions of these samples as suggested above.

Other runs are suggested as follows:

1. The capacity performance characteristics of the distillation unit for a given kettle composition may be determined by varying the rate of vaporization from the still by adjusting the steam valve. From the data obtained curves can be drawn to show the effect of rate of vaporization on composition of distillate, and Murphree Plate efficiency.

2. By varying the reflux ratio and keeping other independent conditions constant, data and curves can be obtained to show the effect of varying reflux ratio on the change of composition per plate, composition of distillate, and hence the reflux ratio and number of plates required to affect a given separation.

3. By varying the kettle composition (still liquid) other runs can be made to show that the same composition of product may be obtained (provided there are a sufficient number of plates in the column) by regulating the reflux ratio.

Kettle compositions can be decreased by discharging the distillate into the receivers instead of the still kettle. When the desired still liquid composition is obtained, establish constant state conditions by discharging the distillate again into the still kettle.

When the last run is completed, discharge distillate into receivers, regulating reflux ratio to obtain, as far as possible, a high composition of distillate in the more volatile component. Continue until the still and the plates contain practically none of the more volatile component.

PRELIMINARY REPORT

By examination and study of the still indicate on a sketch the valves to be opened or closed, points of sampling, and methods of control.

Submit after squad conference, under supervision of the foreman, data sheets for the observations to be made, one for each station and observer.

Discuss in detail the assumptions made in the McCabe-Thiele method. Show by illustrative calculations, using assumed values, the magnitude of error introduced by making these assumptions on the plate to plate change of vapor composition.

Set up detailed material and heat balance equations indicating an alternative method of calculating the change of vapor composition from plate to plate using weight in pounds and weight per cent instead of molal units.

Submit a plot of minimum reflux ratio versus the composition of the liquid in the still for the various runs assigned to your group.

REPORT

Detailed report besides the usual clarifying organization should contain the following:

1. Objectives.
2. All observed and calculated data.
3. Plots of calculated minimum reflux ratio and actual reflux ratio versus the composition of the liquid in the still.
4. Plots of liquid composition versus plate number, as observed, and as calculated by the McCabe-Thiele method using the observed composition of the liquid on the plate below as the basis of computation for each plate.
5. Plots of Murphree plate efficiency versus composition of the liquid on the plate for the various runs made.
6. Plots as suggested under items 1, 2 and 3 (Procedure) if these runs were made.

SAMPLE DATA AND CALCULATIONS

	Run No. 2
Liquid in the still kettle:	
Charge to still kettle. Methanol-water mixture. Pounds	408
Composition of charge. Per cent methanol by weight	39.25
Composition of liquid in the kettle during the run. Per cent methanol by weight	15.00
Composition of liquid in the kettle during the run. Mol fraction methanol	0.091
Product (distillate):	
Product. Pounds per minute	1.1
Composition of product. Per cent methanol by weight	99.2
Composition of product. Mol fraction methanol	0.987
Product. Mols per minute	0.0346
Reflux:	
Reflux. Pounds per minute (by rotameter measurements)	2.00
Reflux. Pounds per minute (heat balance condenser cooling water)	1.97
Composition of reflux. Methanol by weight	99.2
Composition of reflux. Mol fraction methanol	0.987
Reflux. Mols per minute	0.0628

Vaporization:

Temperature of vapors from top plate. Degrees C.	62.2
Vaporization from top plate. Mols per minute	0.0974
Vaporization from top plate. Cubic feet per second	0.715
Superficial velocity top plate. Feet per second	0.668
Vaporization from top plate. Pounds per minute	3.1

Reflux ratio:

Actual reflux ratio (reflux to vapor R/V)	0.6451
Minimum reflux ratio. Calculated from liquid in the still and composition of product (reflux to vapor R/V)	0.627

Steam and water consumption:

Pounds of steam from and at 212° F. per pound of product	1.4
Gallons of condenser cooling water per pound of product	3.61

Calculation of Murphree Plate Efficiency (E_m):

The Murphree plate efficiencies may be calculated by either the graphical or the algebraic method. The algebraic gives more accurate results, in cases where plate to plate changes in composition are small. Both methods are illustrated below.

Graphical Method. The steps in the procedure were as follows:

1. Plotted vapor-liquid equilibrium curve, Fig. 11, of the system methanol and water from data given by Carey.[1]
2. Drew the 45° diagonal as illustrated, Fig. 11.
3. Located point "a" ($x_d = 0.987$ mol fraction methanol) on 45° diagonal. This was one point of the operating line.
4. Located the other point "b" of the operating line by calculating the Y intercept as follows.

The equation of the "operating line" is:

$$y_{n-1} = \frac{Rx_n}{V} + \frac{Dx_d}{V} \tag{10}$$

When $x_n = 0$; the Y intercept is

$$y_{n-1} = \frac{Dx_d}{V}$$

Substituting the values for Run No. 2

$$y_{n-1} = \frac{1.1 \times 0.987}{2.0 + 1.1} = 0.35$$

Therefore the coordinates of the point "b" (the other point of the "operating line") was $x_n = 0$, and $y_{n-1} = 0.35$.

[1] Carey, Chemical Engineers Handbook, ibid., page 1131.

Murphree Plate Efficiencies E_m	Run Number 2		
	Product	Reflux	Liquid in Still
Composition. Per cent methanol by weight	99.2	99.2	15.0
Composition. Mol fraction methanol	0.987	0.987	0.091
Rate of flow. Pounds per minute	1.1	2.0	
Reflux ratio (reflux to vapor R/V) 0.6451			
Y intercept of operating line ($x = 0$). Mol fraction 0.350			

Plate No.	Temp. Liquid on Plate, ° C.	Composition Liquid on Plates (Observed)		Composition of Vapors, Mol Fraction (Calc.)			$y_n - y_{n-1}$	$y_n^* - y_{n-1}$	Murphree Plate Efficiency Factor (E_m)
		Weight, % Methanol	Mol Fraction Methanol	y_n^*	y_{n-1}	y_n			
8	67.8	88.0	0.805	0.927	0.869	0.907	0.038	0.057	66.0
9	69.4	82.75	0.728	0.895	0.820	0.869	0.049	0.075	66.4
10	71.7	74.50	0.622	0.850	0.751	0.820	0.068	0.098	69.8
11	73.9	62.75	0.487	0.790	0.664	0.751	0.087	0.125	71.3
12	76.7	45.75	0.322	0.70	0.558	0.664	0.106	0.142	75.0
13	81.1	30.00	0.194	0.595	0.475	0.558	0.082	0.119	69.3

5. Drew the operating line "*ab*" as shown in Fig. 11.

6. Located the points: 8, 9, 10, 11, 12, and 13 on the operating line. These points corresponded to the observed composition of the liquid on the respective plates in terms of mol fraction methanol.

Each of these points represents two values: "x_n," the composition of the liquid on the plate, and "y_{n-1}" the composition of the vapor from the plate below. For example, point 12, on the operating line "*ab*," represents the composition of the liquid on the twelfth plate, 0.322 mol fraction methanol, and the composition of the vapor (y_{n-1}) from the plate below, the thirteenth plate, 0.558 mol fraction methanol. Points 8, 9, 10, 11, and 13 have similar significance.

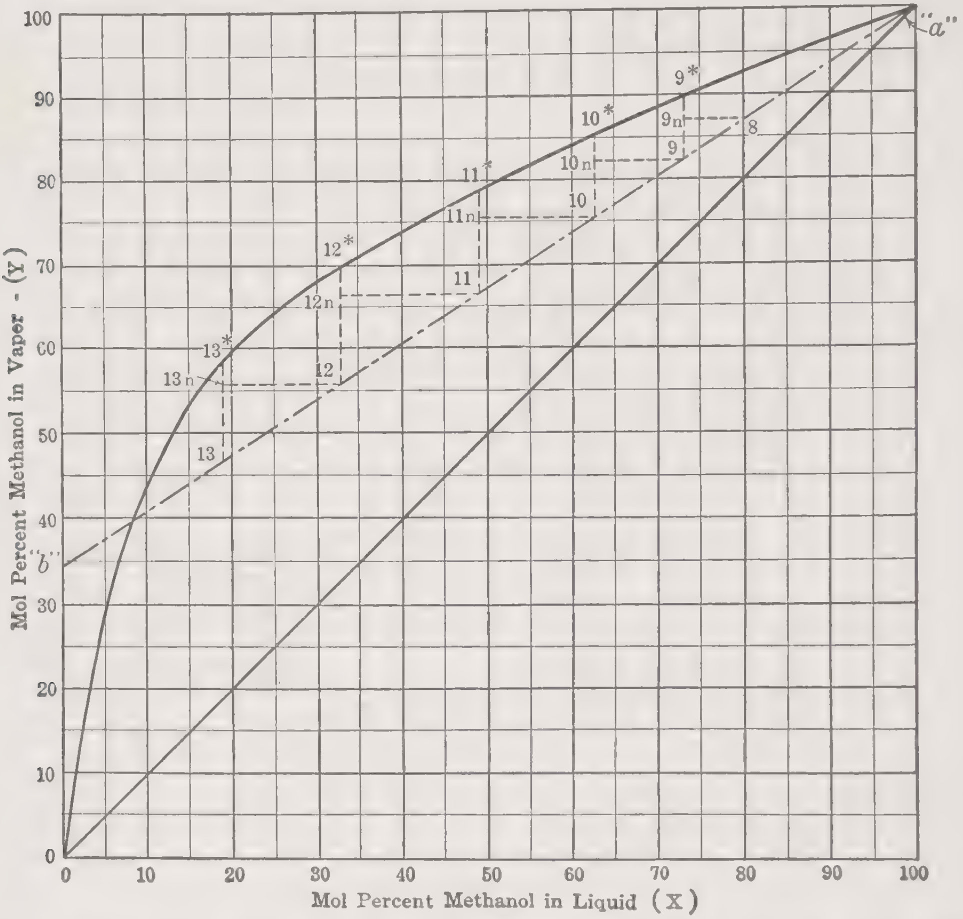

FIG. 11.—Murphree Plate Efficiency. McCabe-Thiele Method, Methanol and Water.

7. Drew the vertical lines: 9-9*, 10-10*, 11-11*, 12-12*, 13-13*. The intersection of these lines with the equilibrium curve give the y_n^* values of the vapors from each of the plates. For example, point 12*, 0.70 mol

fraction methanol, represents what the composition of the vapor from the twelfth plate would have been if the plate was an "ideal plate," i.e., 100% efficiency. The line 12-12* represents the change in vapor composition from the thirteenth (0.558 mol fraction methanol) to the twelfth plate (0.70 mol fraction methanol) under conditions of 100% efficiency, or a change of composition $(y_n^* - y_{n-1})$ of 0.142.

8. Drew the horizontal lines: 8-9_n, 9-10_n, 10-11_n, 11-12_n, 12-13_n. Each of the points as 9_n, 10_n, 11_n, 12_n, 13_n (in terms of the Y coordinate) represent the actual composition of the vapors from the ninth, tenth, eleventh, twelfth, and thirteenth plates, respectively, and determine the actual enrichment of the vapors from plate to plate. For example, point 12_n, 0.664 mol fraction methanol, represents the actual composition of the vapor from the twelfth plate. Therefore the actual change of plate composition from the thirteenth to the twelfth plate was:

$$y_n - y_{n-1} = y_{12} - y_{13} = 0.664 - 0.558 = 0.106$$

It has been shown that under conditions of 100% plate efficiency the change of composition would have been:

$$y_n^* - y_{n-1} = y_{12}^* - y_{13} = 0.70 - 0.558 = 0.142$$

The Murphree plate efficiency of the twelfth plate was therefore found to be:

$$E_{12} = \frac{(y_n - y_{n-1})100}{(y_n^* - y_{n-1})} = \frac{(y_{12} - y_{13})100}{(y_{12}^* - y_{13})} = \frac{(0.664 - 0.558)100}{(0.70 - 0.558)} = 75\%$$

The plate efficiencies of the other plates were computed in a similar manner.

Algebraic Method. Since for a given set of operating conditions the values of R, V, D, x_d, *and* x_n are known from observations made, the value of y_{n-1} may be calculated by substituting the appropriate values in the equation of the operating line:

$$y_{n-1} = \frac{Rx_n}{V} + \frac{Dx_d}{V} \tag{10}$$

For example, in Run No. 2, x_n (plate 12) = 0.322 mol fraction methanol.

Therefore y_{n-1} or y_{13}, the composition of the vapors from the plate below (the thirteenth plate):

$$y_{13} = \frac{2.0 \times 0.322}{3.1} + \frac{1.1 \times 0.987}{3.1} = 0.558$$

In like manner, y_n, or y_{12}, may be calculated from the known value of $x_{11}(x_n)$ of the eleventh plate which was found to be 0.487 mol fraction methanol

$$y_{12} = \frac{2.0 \times 0.487}{3.1} + \frac{1.1 \times 0.987}{3.1} = 0.664$$

y^*_{12}, the composition of the vapor from the twelfth plate under conditions of 100% plate efficiency, was obtained from vapor liquid equilibria data of methanol and water[1] and was found to be 0.70 mol fraction methanol.

Therefore the Murphree plate efficiency of the twelfth plate was found to be:

$$E_{12} = \frac{(y_{12} - y_{13})100}{(y^*_{12} - y_{13})} = \frac{(0.664 - 0.558)100}{(0.70 - 0.558)} = 75\%$$

Calculation of Vaporization from Top Plate. *Cubic Feet per Second:*

$$\text{Vaporization from the top plate} = R + D = 2.0 + 1.1 = 3.1 \text{ pounds per minute}$$

$$\text{Pound mols methanol in these vapors} = \frac{3.1}{32} \times 0.987 = 0.0953$$

$$\text{Pound mols water in these vapors} = \frac{3.1}{18} \times 0.013 = \underline{0.0021}$$

Total pound mols of vapors from top plate per minute = 0.0974

Cubic feet of vapors per second from the top plate

$$= \frac{0.0974(359)(273 + 62.2)}{60 \times 273} = 0.715 \text{ c.f.s.}$$

Superficial velocity from the top plate:

$$\text{Cross sectional area of column} = \frac{3.1416D^2}{4} = \frac{3.1416}{4}(14/12)^2$$

$$= 1.07 \text{ square feet}$$

$$\text{Superficial velocity feet per second from the top plate} = \frac{0.715}{1.07}$$

$$= 0.668 \text{ feet per second}$$

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[1] Carey, Chemical Engineers Handbook, ibid., page 1131.

Calculation of Minimum Reflux Ratio:

Since the system methanol and water has an equilibrium curve of normal shape the minimum reflux ratio was calculated by the equation given below.[1]

$$\frac{R}{V} = \frac{x_d - y_s^*}{x_d - x_s} \tag{18}$$

where R/V = Minimum reflux ratio for a mixture in the still containing x_s mol fraction of the more volatile component

y_s^* = mol fraction of the more volatile component in the vapor in equilibrium with x_s mol fraction of the more volatile component in the liquid in the still

x_d = mol fraction of the more volatile component in the distillate

Substituting the corresponding values in equation 18

$$\frac{R}{V} = \frac{0.987 - 0.425}{0.987 - 0.091} = 0.627 \text{ mol of reflux per mol of vapor}$$

EXPERIMENT NO. 30

Vaporization and Thermal Efficiency of Steam Still

Object. 1. To determine the distillation and thermal efficiency of a live steam still.

2. To compare experimentally determined temperatures of live steam distillation with values computed from vapor pressure data.

Equipment. The type of still that may be used for this problem is shown in Fig. 12. Live steam is admitted into the still by a single coil in which are drilled a series of ⅛ inch holes. The vapors from the still are condensed and cooled in a worm coil, and collected in calibrated receivers, or tarred containers placed on scales.

Rate of distillation may be measured by time-volume, or time-weight observations of the distillate collected in the receivers.

Steam line pressure may be measured by a steam pressure gage placed in the steam line, and just ahead of the live steam control valve of the still.

Procedure. Weigh and charge the material, such as benzol; toluol; carbon tetrachloride,[2] to be steam-distilled into the still. In working with *organic liquids* observe the precautions emphasized in previous problems in regard to *inflammability, air-vapor explosion hazards, and toxicity.*

---

[1] Walker, Lewis, McAdams, and Gilliland, ibid., page 565.

[2] Carbon tetrachloride will eventually corrode copper and iron parts of the still through hydrolysis.

Provide adequate receiving vessels. Allow a substantial amount of water to flow through the condenser and adjust to maintain the desired temperature after distillation begins.

If operating instructions specify distillation to be conducted under vacuum, regulate vacuum pump so that the desired vacuum is obtained.

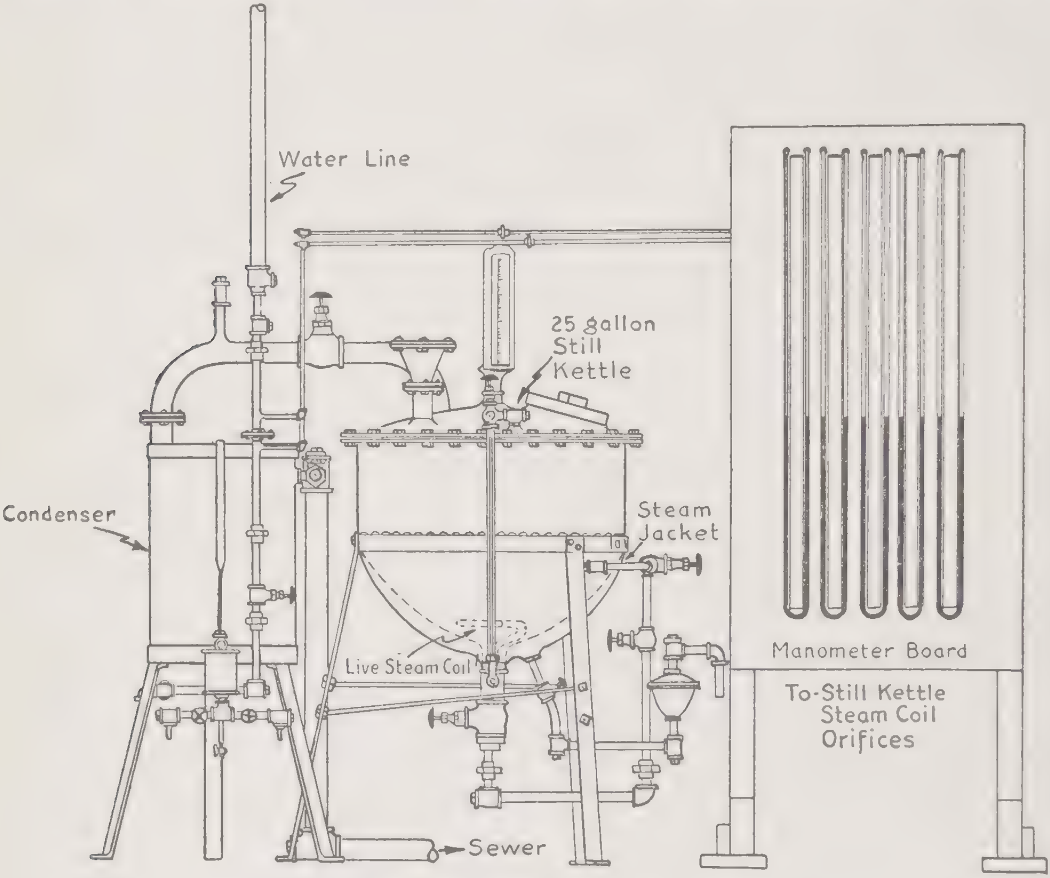

Fig. 12.—Live Steam Still.

Crack the steam valve and slowly open to obtain the desired rate of distillation as specified by instructions.

Shut off steam valve and discontinue distillation when the receivers indicate that the desired amount of charge, as specified by instructions, has been distilled over.

Separate the water from the material being distilled in both the distillate and residue in the still kettle by decantation. Determine the amount of each by weighing.

Make other runs as specified, varying the rate, and the pressure of distillation.

PRELIMINARY REPORT

By examination and study of the still indicate on a sketch the valves to be opened or closed, points of sampling, and methods of control.

Submit, after squad conference, under supervision of the foreman, data sheets for the observations to be made, one for each station and observer.

Show by application of the phase rule why the temperature of distillation, and the composition of the product should remain constant in the steam distillation of a liquid which is completely immiscible in water.

Show by application of the Clausius-Claperyon equation [1] the effect of change of pressure under which steam distillation is conducted on the amount of steam required for (a) materials with a low vapor pressure, and (b) materials with a high vapor pressure. Illustrate by actual numerical calculations.

Show how a Mollier Diagram may be used in steam distillation calculations to predict the temperature of steam distillation under given pressure conditions for the special case in which superheated or high pressure steam is used, and condensation of steam is not permitted to take place in the still kettle. By calculations compare the steam required for this case against the steam required under ordinary steam distillation conditions, and at the same pressure.

REPORT

Detailed report, besides the usual clarifying organization, should contain the following:

1. Objectives.
2. All observed and calculated data.
3. Plots of distillation and thermal efficiency versus rate of distillation.
4. Plot of steam required versus pressure of distillation, observed and calculated.

SAMPLE DATA AND CALCULATIONS

	Run No. 3
Charge to the still. Pounds of toluol	43.0
Pressure in still during run. Absolute mm. Hg.	754
Temperature of steam distillation. Degrees C. (experimental)	84
Temperature of steam distillation. Degrees C. (calculated)	84.2
Pounds of water in distillate per pound of toluol (experimental)	0.237
Pounds of water in distillate per pound of toluol (calculated)	0.245

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[1] Sidgwick, Trans. Chem. Soc., 117, page 396 (1920).

	Run No. 3
Vaporization efficiency in steam distillation	103%
Steam line pressure. Pounds per square inch (gage)	40
Quality of steam. Per cent steam	97
Total amount of steam used. Pounds	18.5
Total amount of toluol steam distilled. Pounds	32.0
Pounds of toluol in the still at the end of the run	10.45
Total equivalent amount of steam required to heat unvaporized toluol to temperature of steam distillation	0.3
Actual amount of steam used in distillation	18.2
Pounds of steam used per pound of toluol steam distilled	0.568
Pounds of steam required (calculated), assuming no heat losses and 100% vaporization efficiency in steam distillation	0.418
Thermal efficiency of steam distillation. Per cent	73

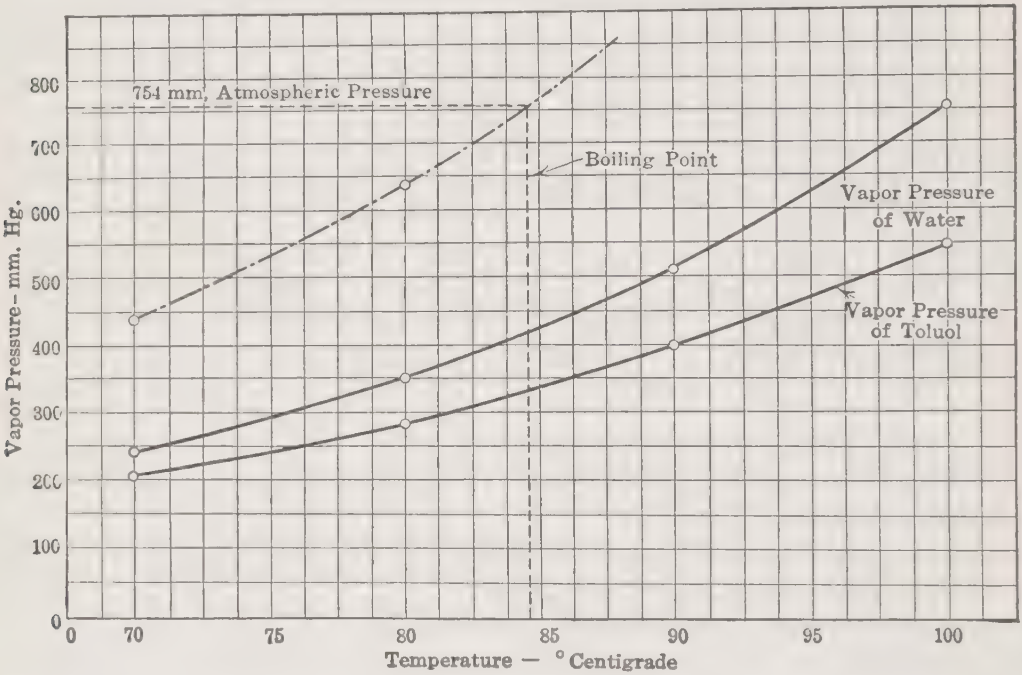

FIG. 13.—Vapor Pressure Curves for Steam Distillation. Calculations Toluol-Water.

Temperature of Steam Distillation:

The steps in the calculation were as follows:

a. Plotted vapor pressure of toluol, and water versus temperature as illustrated in Fig. 13.

b. Since the total pressure exerted by a mixture of immiscible liquids is the sum of the vapor pressures of the individual components, the total vapor pressure curve, Fig. 13, was drawn through points computed by adding the vapor pressure of water at a particular temperature to the vapor pressure of toluol at the same temperature.

c. Since the distillation was conducted under 754 mm. Hg pressure the boiling point was noted as indicated in Fig. 13 from the intersection of the 754 mm. Hg isobar with the total pressure curve and was noted to be 84.2° C.

Pounds of Water per Pound of Toluol in Distillate:

This was computed from the equation relating the weights of the two components in the distillate with their respective molecular weights and vapor pressures at the temperature of steam distillation.

$$\frac{W_a}{W_b} = \frac{P_a M_a}{P_b M_b} = \frac{420 \times 18}{335 \times 92} = 0.245 \text{ pound of water per pound of toluol in the distillate}$$

Vaporization Efficiency in Steam Distillation:

This may be calculated by the equation given by Carey[1]

$$E_v = p/p^* \tag{19}$$

where E_v = vaporization efficiency in steam distillation
p = actual partial pressure of volatile material in steam
p^* = partial pressure of volatile component which would be in equilibrium with the liquid charge.

Equation (19) may also be written in this form:

$$E_v = \frac{W_e}{W_c} \times 100 \tag{20}$$

where W_e = actual pounds of toluol in the distillate per pound of water, as observed from experimental data, and which, for Run No. 3 was 1/0.237 pound of toluol per pound of water
W_c = calculated pound of toluol in the distillate per pound of water = 1/0.245

Therefore

$$E_v = \frac{W_e}{W_c} = \frac{0.245}{0.237} \times 100 = 103\% \text{ vaporization efficiency in steam distillation}$$

Thermal Efficiency of Steam Distillation:

This item may be defined mathematically by the following equation:

$$E_t = \frac{W_s \times 100}{W_u} \tag{21}$$

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[1] Carey, Chemical Engineers Handbook, ibid., page 1166.

Where

W_s = Calculated pounds of steam required per pound of toluol distilled

W_u = Actual pounds of steam used, observed from experiment, per pound of toluol distilled

The thermal efficiency, E_t, was computed from a heat balance equation using as a basis 1 pound of toluol, and a reference temperature of 32° F. and solving for W_s, the pounds of steam required under conditions of 100% vaporization efficiency in steam distillation and assuming no heat losses.

HEAT BALANCE CALCULATIONS

Input

a. *Heat content of charge* = $1 \times 0.40 \times (80 - 32) = 19.2$ B.t.u. Where 80° F. was the temperature of the charge, and 0.40 the average specific heat of toluol as given by Hougen and Watson.[1]

b. *Heat content of steam used* = $W_s \times 1148.3 = 1148.3\ W_s$. Where 1148.3 was the total relative enthalpy of steam at 40 pounds per square inch gauge pressure and 97% steam quality obtained from Mollier diagram.[2]

Total heat input = $1148.3\ W_s + 19.2$.

Output

a. *Heat content of toluol vapors* = $1 \times 195.4 = 195.4$ B.t.u. Where 195.4 was the total relative enthalpy of toluol vapors at 183.5° F. It is the sum of the latent heat of vaporization of toluol at 183.5° F. and the heat of the liquid (above 32° F.) of toluol at the same temperature. The heat of the liquid was calculated from the average specific heat of toluol as a liquid, and was found to be 31.9 B.t.u.'s per pound of toluol, while the latent heat of vaporization of toluol at 183.5° F. was calculated by the method given by Hougen and Watson,[3] and was found to be 163.5 B.t.u. per pound of toluol.

b. Heat content of water vapor: = $0.245 \times 1137.4 = 279$ B.t.u. Where 1137.4 is the total relative enthalpy of steam at 183.5° F (above 32° F.) as obtained from Keenan and Kayes Mollier Diagram.

c. Heat content of condensed water in still: = $(W_s - 0.245) \times 147.8$. Where 147.8 is the total relative enthalpy of water above 32° F. at 183.5° F.

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[1] Ibid., page 119.

[2] Mollier Diagram. Keenan and Kayes Steam Tables, ibid.

[3] Ibid., page 140.

Total Heat Output: = 195.4 + 279 + ($W_s - 0.245$) 147.8.

Equating heat input and output:

$1148.3\, W_s + 19.2 = 195.4 + 279 + (W_s - 0.245)\, 147.8.$

$W_s = 0.418$ pound of steam required per pound of toluol vaporized as distillate.

Actual pounds of steam used per pound of toluol distilled. Since all the toluol charged to the still was not distilled a correction must be made for the toluol heated to the distillation temperature but not distilled.

Steam required to heat unvaporized toluol to 183.5° F.

$$\frac{10.45 \times 0.42(180 - 100)}{1148.3} = 0.3 \text{ pound}$$

$$W_u = \frac{\text{Total pounds of steam used} - \text{Correction for unvaporization}}{\text{Total pounds of toluol distilled}}$$

$$= \frac{18.5 - 0.3}{32} = 0.568$$

$$E_t = \frac{W_s \times 100}{W_u} = \frac{0.418}{0.568} = 73\%$$ thermal efficiency of steam distillation.

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CHAPTER VI

THE DRYING OF SOLIDS

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The drying of solids refers to the subtraction of a liquid from a solid phase as in the removal of water from textile, wet clay, crystals of salt, soap, and lumber. The removal of liquids other than water is also considered as a drying operation. Under this broad definition of drying are included the removal of a liquid by a hydraulic press, as applied to the pressing out of a solvent from extracted oil cake; the removal of excess water from wet crystals or textiles by a centrifugal basket; the scraping of water from leather as in hand setting and staking; the withdrawal of water from wet clay objects by a plaster of paris mold; the wiping of a liquid from a surface by the capillary action of a towel; the drying of lumber by a current of heated dry air; and the evaporation of water from soap chips by applying heat under conditions of reduced pressure or in a vacuum drier. The first four methods of drying are mechanical and will be considered properly elsewhere in this text under the operations of mechanical separation by pressure, by centrifuging, and by capillarity. The last two methods will be considered separately under the divisions of *air drying* and of *vacuum drying.* In both air and vacuum drying the capillary flow of liquid must be considered as well as the removal of the liquid by vaporization.

AIR DRYING

Air drying refers to the removal of a liquid from a solid by vaporization when in contact with an atmosphere of unsaturated air.

Definitions. A few definitions are first in order. By *constant drying conditions* are meant that the velocity and direction of air, its average temperature, pressure and humidity are constant, and that the method of supporting and exposing the stock is uniform and fixed. *Equilibrium moisture content* represents the moisture bound by the stock, due to the depression of the normal vapor pressure by the presence of soluble materials or by the great surface energy of the stock, such as in fibrous or highly porous solids. Equilibrium moisture content represents that held by the solid in equilibrium with air of definite temperature, humidity and pressure. Two values of equilibrium moisture content are manifest, one for sorption and the other for desorption; the latter value is higher than the former. In drying problems, the desorption values are of particular interest. Equilibrium moisture content of a solid increases very irregularly with increase in relative humidity and decreases slightly with an increase in temperature. (Fig. 1.) *Free moisture* content represents the difference between the total and equilibrium values. For convenience in calculations, moisture contents are expressed in pounds of water on the basis of one pound of dry stock. Air drying usually proceeds in several stages, classified according to the rate of drying under constant drying conditions, namely, *a uniform rate*, a *first falling rate*, and a *second falling rate period*. The transition from one rate of drying to another is referred to as a critical point and the corresponding average moisture content as an average critical moisture content. These critical moisture contents, however, are not fixed values for any given material but depend upon the thickness of stock and upon the rate of drying. *Fiber saturation* point refers to the equilibrium moisture content as the humidity of the air approaches saturation.

Theory of Capillary Flow of Moisture in a Granular Solid.[6] In a granular solid the flow of moisture resulting from absorption, drainage or evaporation is induced by gravitational and capillary forces. When the pore space within a granular solid is only partly filled with water, a suction is produced which depends upon the surface tension of the liquid and the radii of curvature of the air-water interface. For an air bubble within a liquid the suction tending to collapse the bubble is $2T/r_2$. For a liquid nodoid of revolution held between two grains the suction force is equal to $T\ (1/r_2 - 1/r_1)$ dynes per square centimeter, where r_1 is the radius of curvature of the convex surface in centimeters, r_2 is the radius of the concave surface, and T is the surface tension of the liquid in dynes per centimeter. Within an unsaturated granular solid the liquid will move in the direction of increasing liquid curvatures under the force of capillary suction. When evaporation of water takes place from the top surface of a horizontal layer of a granular solid resting in a pan and starting with saturated pore spaces the water surface is at first depressed into the branched opening among the grains on the top surface. The curvature developed in the water surface produces a suction. Any attempt to lift the entire column of water bodily through the sand is resisted by the weight of the water column and by the suction produced at the bottom surface when a curvature is formed there. The suction required to blow a bubble of air into a branched opening of liquid among the grains is equal to $2T/r_2$ dynes per square centimeter, and is called the *entry suction*. For uniform spheres in closest packing this *entry suction* is $12.9\ T/r$ dynes per square centimeter, where r is the radius of the spheres. For grains irregular in shape and size, the entry suction is much less than 12.9 T/r. The entry suction will increase as successively smaller pores are opened. The development of suction due to evaporation will draw more water to the surface against the force of gravity. Water will continue to be drawn to the surface until the suction produced is insufficient to hold up, against gravity, the increasing column of water, then a continuous film is no longer maintained. The water will then break into detached nodoids of revolution about points of contact between grains. Evaporation will proceed from these nodoids, increasing the thickness of dry grains at the surface with the level of evaporation retreating below the surface of the solid.

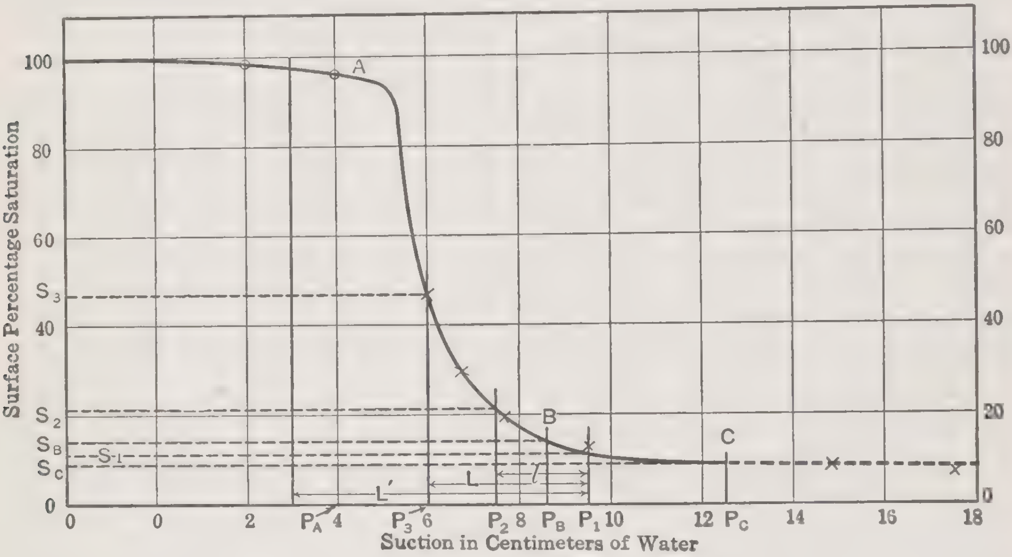


FIG. 1.—Suction Saturation of No. 3.5 Sand.[6]

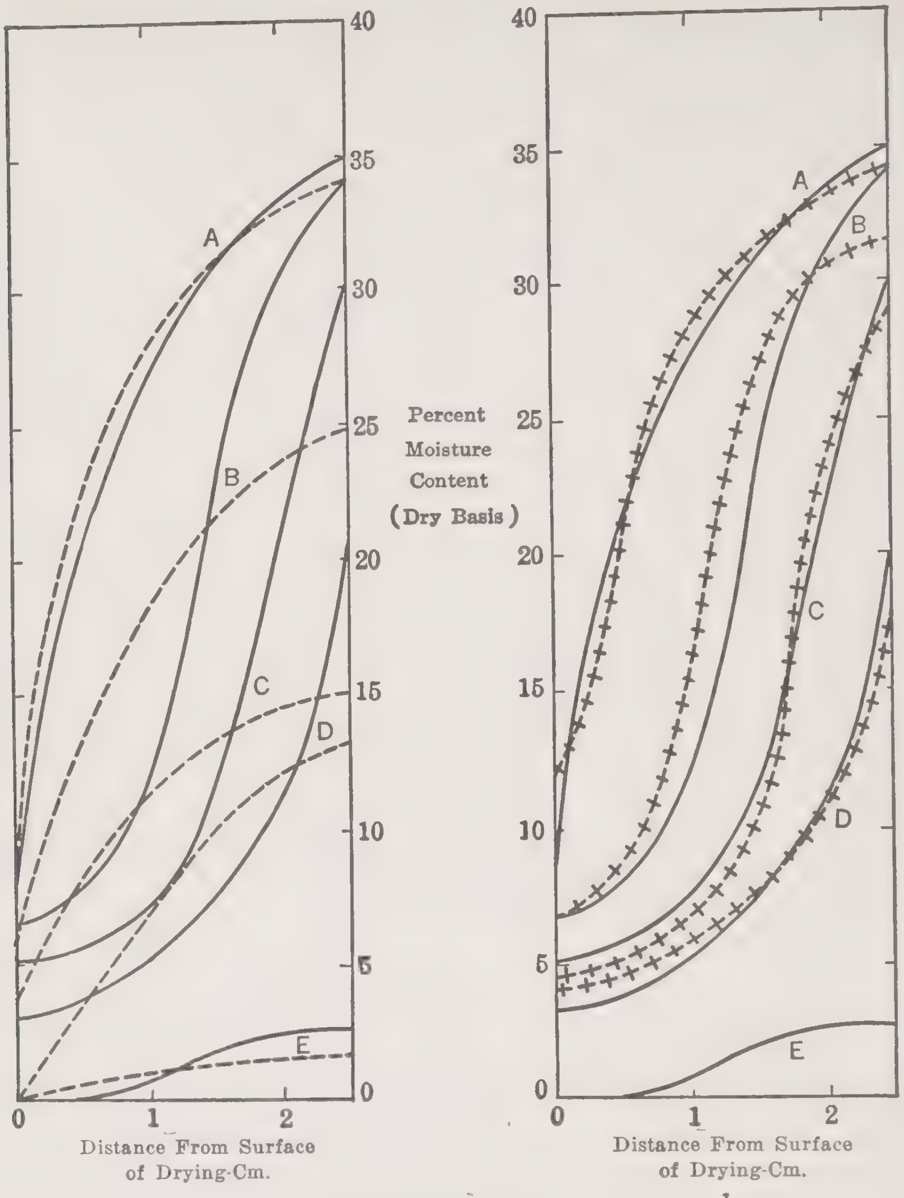
In Fig. 1 is shown the relation of suction in centimeters of water to the percentage saturation of a certain sand at the surface. The entry suction P_A corresponds to 4 cm. of water, point A. For a horizontal layer of sand drying only from the top surface, the water distribution can be calculated for any thickness and for any average water content. For positions in the sand below values of the entry suction P_A saturation should be assumed. For values above 12.5 cm., point C, increased suction does not withdraw further liquid water to the surface. At this stage the pendular state of water distribution exists corresponding to a saturation of 9%.

The moisture distribution in a granular solid can be calculated as follows when evaporation is taking place from the top surface only and when the relations of Fig. 1 are fulfilled. For a suction of P_1 cm. at the surface the percentage moisture saturation at the surface is S_1. For a distance l cm. below the surface the percentage saturation can be obtained by measuring the value S_2 from the graph at $P_1 - l$ cm. For a total thickness of sand of L cm. the percentage saturation at the bottom is S_3 cm. For positions extending above the entry suction A 100% saturation should be assumed rather than the value on the graph. For example, with a layer L' cm. thick, the bottom of the sand is fully saturated. After plotting the moisture distribution over the entire thickness of the sand layer the average moisture content can be obtained by graphical integration. The moisture distribution, calculated by this method, is shown in Fig. 2, compared with experimental values. This method cannot be extended after the surface reaches the pendular state, that is, beyond point C. In drying beyond this point the water film starts to break at the top surface where the concentration is least. The broken nodoids of water will then dry by evaporation *in situ* producing a layer of complete dryness at the surface. The thickness of complete dryness increases as drying continues. An examination of *a* and *b* in Fig. 2 shows that the moisture distribution in a granular solid during drying is extremely complex, changing in inflection and curvature as drying proceeds. The moisture distribution, as calculated from diffusional equations, shows a non-inflected curve as in Fig. 2, *b*. These facts indicate that the flow of water in a granular solid is not a diffusional process, as often assumed, but rather a capillary one. The actual moisture distribution depends upon suction-saturation relationships which in turn are dependent upon the size and distribution of particles and pores. These relations can be arrived at experimentally but the mathematical approach has not been fully formulated.

Drying a Granular Solid

The actual mechanism involved in the air drying of a solid is involved and complicated and as yet no general formulation has been developed suitable for all materials. The first case to be considered is the drying of a horizontal layer of a granular solid such as sand placed in a tray with drying from the top surface only It will be assumed that constant drying conditions prevail, and that all the heat received by the solid enters the top surface only and entirely from the air with no gain of heat by radiation and conduction except from the air. It will also be assumed that the wet stock is initially saturated, with no air spaces, at the wet bulb temperature of the air, and that all the moisture is free.

Constant Rate Period. In the constant drying rate period the surfaces of grains at the top surfaces remain completely wetted even though the moisture content is steadily diminishing. The fraction of wetted surface of the grains remains constant even though the pores are gradually



a *b*

FIG. 2.—Moisture Distribution during Drying of Sand.[6, 14]

Experimental ————————

a. Calculated from diffusion equation — — — — —

b. Calculated from capillary suction + + + + +

opened and depleted of their water content. The flow of water by capillarity is sufficient to maintain a uniformly wetted surface during this period. The cells below this surface open when their entry pressure is attained. The suction at the surface steadily increases by an amount equal to the gravity head from surface to saturation level. A stream of water reaches the surface, sufficient to maintain a constant wetted area and a constant evaporation rate. When the flow of water by capillarity can no longer keep pace with the initial rate of drying, the fraction of wetted area at the top surface gradually diminishes with a corresponding decline in drying rate. The water at the surface then begins to recede to higher and higher curvatures, the wetted area begins to diminish as the water is pulled in between the grains; the *first falling rate period* then begins.

The end of the constant rate period occurs at a definite suction value at the surface, corresponding to a definite surface moisture content. This point is referred to as the *first critical point* and corresponds to a definite moisture content at the surface but does not correspond to a definite average moisture content, since the average moisture content corresponding to the first critical point increases with the thickness of the layer and with the rate of drying.

The temperature of the grains remains nearly constant during the constant drying rate period. The rate of evaporation remains constant because the wetted area, temperature, vapor pressure and drying conditions are all constant.

The rate of drying per unit area of top surface during the constant rate period can be expressed by the equation

$$-\frac{dW}{Ad\theta} = k(p_s - p_a) \tag{1}$$

where W = water evaporated
θ = time elapsed
A = area
p_s = vapor pressure of the water at the surface
p_a = partial pressure of water vapor in the air
k = drying coefficient as defined by Equation (1)

Since Δp is nearly proportional to the difference in humidity of the air, Equation (1) may be written

$$\frac{dW}{Ad\theta} = k' \, (\Delta H) \tag{2}$$

where $\Delta H = H_s - H_a$.

Where drying proceeds at the wet bulb temperature of the air $\Delta H = H_w - H_a$, where H_w is the humidity of air at its wet bulb temperature and ΔH is the unsaturation of the air during adiabatic drying.

The rate of drying per unit area of surface during the constant rate period is dependent only upon drying conditions and not upon thickness of layer. The coefficient k' is hence dependent upon the velocity and direction of air flow and the position of the surface. Increase in temperature increases H_s and hence the rate of drying.

Interest is usually confined to the rate of drying based upon one pound of dry stock rather than upon unit surface area. From a weight balance

$$W = (w_i - w)D \tag{3}$$

where W = water evaporated, in pounds
D = weight of dry stock, in pounds
w_i = initial average water content, pounds per pound of dry stock
w = average water content at any given time

Let V = volume occupied by D pounds of dry stock
A = evaporating area when drying from one side for D pounds of dry stock
L = thickness of stock

Then $D = \rho V = \rho AL$, where ρ is the density of the dry stock. (4)

During the constant rate period Equation (2) becomes, when combined with Equations (3) and (4),

$$\frac{dw}{d\theta} = -B\Delta H \tag{5}$$

where $B = -\dfrac{k'}{\rho L}$

The drying coefficient B depends upon all the variables which determine constant drying conditions. It increases with an increase in the velocity of the stock, because of reduced thickness of air film at the interface, and decreases directly with thickness of stock.

Where the water evaporates, at the wet bulb temperature of the air, $\Delta H = H_w - H_a$ and at low temperatures

$$\Delta H = .000238(t_a - t_w) \tag{6}$$

as can be seen from the humidity chart, where ΔH is measured in pounds of water per pound of dry air and t is measured in degrees Fahrenheit.

First Falling Rate Period. When the suction at the surface exceeds the required entry pressure, the rate of drying falls off linearly with the moisture content of the grains. When the suction exceeds this value, water is drawn up into nodoids of higher curvature and the actual wetted area at the surface diminishes.

The rate of drying during the first falling rate period depends upon the same conditions as for the constant rate period and in addition falls off with decreasing moisture content. The rate of drying during the first falling rate period can often be expressed by the equation

$$\frac{dW}{A d\theta} = kw\Delta p \tag{7}$$

where w is the average moisture content of the stock and k is defined by the equation. The drying coefficient k increases with temperature and with the velocity of air. During this stage of drying the temperature of the solid steadily rises with the temperature gradient increasing toward the heating surface. Equation (7) holds only under the condition that the fraction of wetted surface area remain proportional to the average water content of the stock. This condition will obviously not prevail when the stock is so thick that full saturation of pore spaces exists remote from the drying surface at the end of the constant rate period.

Where drying is based upon one pound of dry stock, Equation (7) becomes

$$\frac{dw}{d\theta} = -B'w(\Delta H) \tag{8}$$

where B' is a drying coefficient as defined by Equation (8).

Second Falling Rate Period. The first falling rate period ceases when the curvatures at the surface become so great as to break the continuity of the water film, the surface becomes dry and the dry region extends progressively below the surface of the grains. The second falling rate period now begins. The pendular state exists near the surface. Evaporation takes place from isolated nodoids and hence the evaporating surface retreats below the surface of the solid. Drying hence proceeds by diffusion of vapor through the dried portion of the solid and through the surface air film. The rate of drying during this period is no longer affected by air velocity but increases markedly with increased coarseness of grain. During this period the temperature rises and approaches finally the temperature of the moving air stream. The rate of drying rapidly falls off to zero as the entire layer of grain becomes dry. This slow drying rate period extends over greater and greater average moisture contents as the thickness of grain increases and also for a given moisture content takes a much lower value as the thickness increases.

Distribution of Moisture. As explained previously the distribution of moisture during drying cannot be calculated from diffusion equations but depends entirely upon the complex suction-water concentration relationships of the substance. This relationship is dependent upon particle size, degree of packing, and distribution of pore spaces. The flow of water is determined by capillary forces and is not dependent upon moisture concentrations, indeed, the flow of water may be in the direction of increasing concentrations. If a fine grain layer is placed on top of a coarse grain layer the coarse grain layer dries out faster than the top layer even though evaporation takes place only at the top surface. These facts are all contrary to the requirements of a diffusional process.

Other Factors in Drying. The mechanism of the air drying of a granular substance in a horizontal layer from the top surface only has been described. For drying from the bottom surface it must be recognized that gravity aids the flow by suction to the bottom surface and retards it to the top surface. When drying fibrous materials it must be recognized that free moisture only is removed by evaporation. Due to the large extent of surface and the high curvature between micelles, much water is held bound despite the unsaturation of the air.

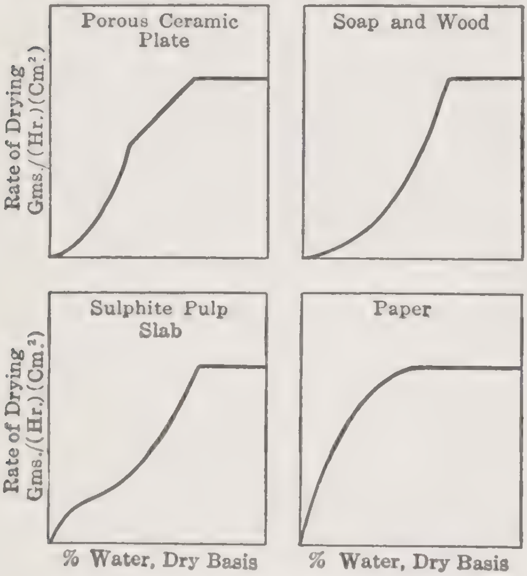


Fig. 3.—Typical Drying Rate Curves. (Sherwood.)[28]

With colloidal or fibrous materials, the resistance to the flow of water by capillarity is so high as not to maintain the usual suction-water concentration equilibria. Under these conditions where the cell structure is fine and evenly subdivided, as in clays and in wood, the rate of drying seems to follow the results obtained by integration of diffusion equations.

Figure 3 illustrates the rate of drying curves as obtained by T. K. Sherwood [28] for four different cases of drying.

Air Drying of Fibrous Materials in Thin Sheets

In the drying of a fibrous material the desorption equilibrium moisture content must be considered since only the free moisture is subject to evaporation. The capillary forces within a fibrous material are so high as to permit neglect of the gravitational force at least in the final stages of drying. It will be assumed that constant drying conditions prevail, that drying is taking place from both sides of the sheet, and that shrinkage is negligible.

In the constant rate period Equation (5) still applies except that now $B = -2k'/\rho L$ since evaporation is taking place from two surfaces instead of one. In the first falling rate period the rate of drying is proportional to the average free moisture content, and Equation (8) becomes

$$\frac{dw}{d\theta} = -B_1 F \Delta H \qquad (9)$$

where F is equal to the free moisture content and is equal to the total moisture content w less the equilibrium moisture content E or $F = w - E$. The equilibrium moisture contents E are desorption values which increase with increasing humidity and decrease slightly with temperature (Fig. 4). Where E is small compared to w, Equation (9) becomes

$$\frac{dF}{d\theta} = -B_2 F \Delta H \qquad (10)$$

The rate of drying during the second falling period is complicated by the increasing thickness of dry solid through which vapor must diffuse, and by the gradually increasing temperature of the stock and non-uniformity of temperature.

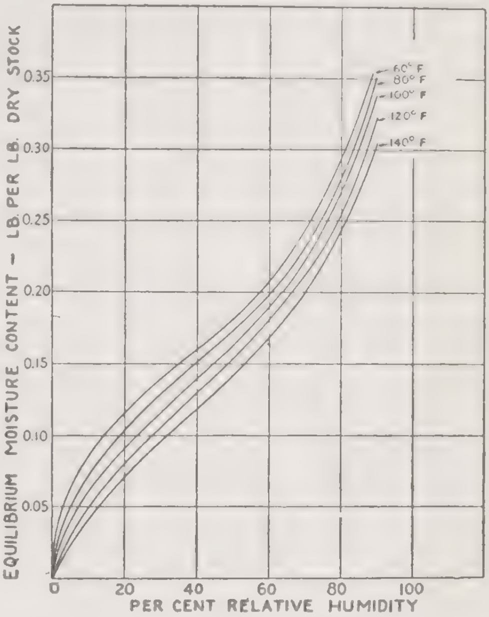


FIG. 4.—Equilibrium Moisture Content of Chrome Leather.

Generalized Method for Constant Rate Period

Shepherd, Hadlock and Brewer[23] have developed a generalized method of calculating the rate of evaporation, during the constant rate period for drying in trays, of non-hygroscopic materials in a current of air. They also showed that more reliable results could be obtained by calculating rates of drying from heat transmission coefficients than from mass transfer coefficients. A drying chart calculated from experimental data on heat transmission coefficients for convection, after correcting for radiation, is shown in Fig. 5. This chart is recommended for the drying of non-hygroscopic materials during the constant rate period. The chart is based upon an air velocity of 5 feet per second; for other velocities the value of R' obtained from the chart should be multiplied by $\left(\frac{u}{5}\right)^{0.8}$ where u is the air velocity in feet per second.

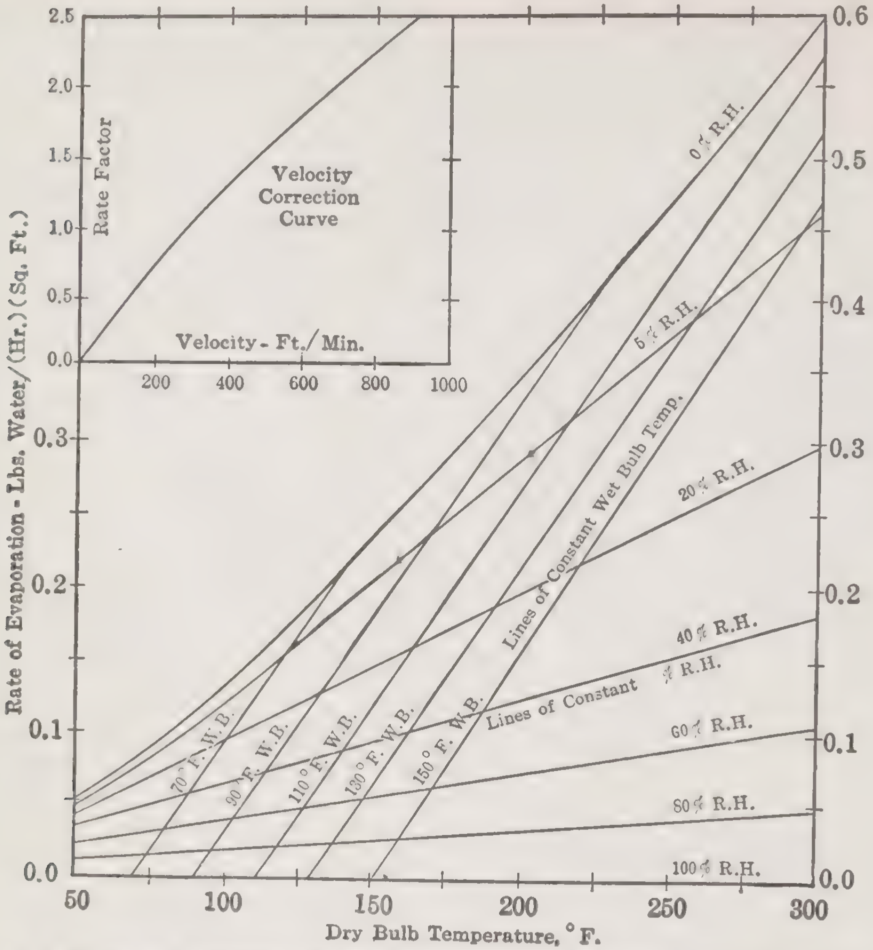


Fig. 5.—Rate of Evaporation Chart from Shepherd, Hadlock and Brewer.[23]

When heat flows into the bottom of the tray as well as through the drying surface, the values obtained for constant wet bulb temperature drying should be multiplied by the factor

$$\left[1 + \frac{1}{1 + \dfrac{h_c L}{k}}\right]$$

where the transfer of heat by radiation and the resistance of the metal pan are negligible, and where the unwetted area of tray is equal to the wetted area of the surface where evaporation is taking place.

h_c = heat transmission coefficient, B.t.u. per hr., sq. ft. °F.
L = thickness of stock, ft.
k = thermal conductivity of stock, B.t.u. per hr. ft. °F.
R' = rate of evaporation, pounds of water per hr. sq. ft.

DRUM DRYING OF A SHEET MATERIAL

The rate of drying a sheet material on an internally steam heated metal drum is determined chiefly by the rate of heat transmission into the sheet from the drum. This method of drying is unique in that the stock is at a higher temperature than that of the surrounding air, so that heat is being transferred *to* the air rather than *from* the air as in the usual case of air drying.

For removal of free moisture during the constant rate period the rate of drying may be obtained from Fig. 10a with which the following equation agrees:

$$R' = 2.45u^{0.8}\Delta p \;/L$$

where R' = rate of drying, pounds per hr./sq. ft.
u = velocity of air passing over surface of sheet, ft. per sec.
$\Delta p = p_s - p_a$, atmospheres.
p_s = vapor pressure of water at outer surface of sheet, atmospheres.
p_a = partial pressure of water vapor in main air stream, atmospheres.

The temperature and hence the vapor pressure at the outer surface of the sheet material may now be obtained from heat transmission data. Resistance to the flow of heat is offered by the condensate on the inside of the drum, by the metal wall, by the sheet material, and by the air film on the outer surface of the sheet. Heat flows to the outer surface of the sheet through the first three resistances in series and into the air stream from the outer surface by three paths in parallel, namely, by convection, by radiation, and by evaporation.

The rate of heat flow is hence given by the equation

$$\frac{Q}{A\theta} = \frac{t_v - t_a}{\dfrac{1}{h_w} + \dfrac{1}{h_m} + \dfrac{1}{h_p} + \dfrac{1}{h_c + h_r + h_c}}$$

where Q = heat flow in B.t.u.
A = area of outer surface of sheet, sq. ft.
θ = time elapsed, hrs.
t_v = temperature of steam inside drum, °F.
t_a = temperature of main air stream, °F.
h_w = heat transmission coefficient of condensate, B.t.u. per hr. sq. ft. °F.
h_m = heat transmission coefficient of metal wall
h_p = heat transmission coefficient of sheet material
h_c = heat transmission coefficient by convection from outer surface

h_r = heat transmission coefficient by radiation from outer surface
h_e = heat transmission coefficient by evaporation from outer surface

Where drying proceeds at a constant rate,

$$h_e = \frac{2.45u^{0.8}\Delta p\lambda}{\Delta t_f}$$

where λ = latent heat of vaporization of water at t_s, B.t.u. per pound
$\Delta t_f = t_s - t_a$
t_s = temperature of outer surface of sheet, °F.
t_a = temperature of main air stream, °F.

EXPERIMENTS ON AIR DRYING

With the exception of fractional distillation no laboratory experiments in chemical engineering illustrate so many important physico-chemical principles and include so many other auxiliary unit operations as do the experiments on air drying, particularly of fibrous materials. In these experiments are involved an understanding of the

mechanism of drying in its various stages,
equilibrium moisture content,
free moisture content,
diffusion of vapors,
capillary flow of liquids,
transmission of heat,
flow of gases and power requirements,
humidity chart with use of wet and dry bulb temperatures,
humidification and dehumidification,
adiabatic vaporization,
heat and weight balances,
economic design for optimum air velocity, air recirculation and size of dryer.

The following experiments in air drying deal with cabinet air driers. All the data desired with respect to progressive driers can be obtained in a cabinet type.

It is particularly important in selecting a laboratory air drier of the cabinet type that great flexibility in operation be secured so that such variables in operation may be studied as temperature, humidity, recirculation of air, and velocity of air flow. In particular it is important that automatic temperature control be provided for any experiment where important results are to be obtained. It is convenient to arrange the drying chamber for both horizontal trays as well as for vertical suspension of sheet materials.

Equipment. Many excellent air cabinet driers suitable for experimental purposes appear on the market. In Fig. 6 is shown a photograph of a representative air cabinet drier with horizontal shelves. In Figs. 7 and 8 are shown the scheme of air circulation in two types of commercial driers. Fig. 9 shows a special adaptation of a tray drier arranged with an overhead scale for weighing the stock while in the drier. The equipment required for experiments on air drying include the following:

Air cabinet drier (laboratory size) is self-contained, with shelves, heating coils, fan for circulation of air, dampers for control of recirculation of air, fan motor. Baffle arrangement is provided for control of velocity over single or over all trays.

A temperature controller operating a throttling valve is desired on the steam supply. Any temperature control which demands complete opening or closing of valve for the entire steam supply will result in wide temperature variations and cannot be considered satisfactory for use in obtaining experimental data of importance.

Variable air velocity should be made possible by control of speed of fan by using either a variable speed motor or a variable pulley ratio on the fan drive. Motors directly connected to fan are undesirable unless a variable speed motor is provided. Without control of air velocity this important variable cannot be studied.

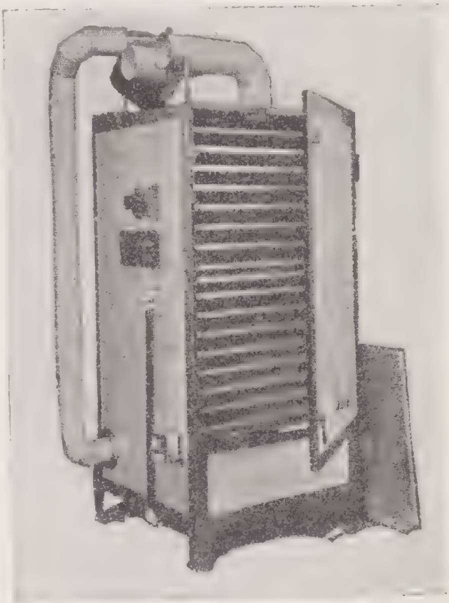


FIG. 6.—Stokes Drying Closet.

Electrically heated with automatic temperature control.

It is desirable to use the same drier for drying in pans and in sheets. Rigid sheet material such as fiber board can be dried horizontally, inserted in place of trays. Flexible sheets must be dried vertically.

It is convenient for experimental purposes to make provision for suspending the entire load within the drier from an outside support, resting on a platform scale or sensitive torsion balance, with provision made for free motion of supporting guides without friction. The platform scale should be placed on top of the drier. With this provision the stock being dried can be directly weighed during any moment of the drying period without opening doors and interrupting the process by removing stock for weighing. For accurate work this is an essential requirement.

Wet and dry bulb thermometers should be placed in the air stream at entrance and exit to stock and arranged for external reading at regular time intervals. An automatic temperature and humidity recorder is desirable.

The velocity of air passing over the stock can be measured, at various points, with an anemometer, or the average velocity can be calculated from a weight balance of water evaporated, measured by direct weighing, and from changes in the humidity of the air in passing over the stock.

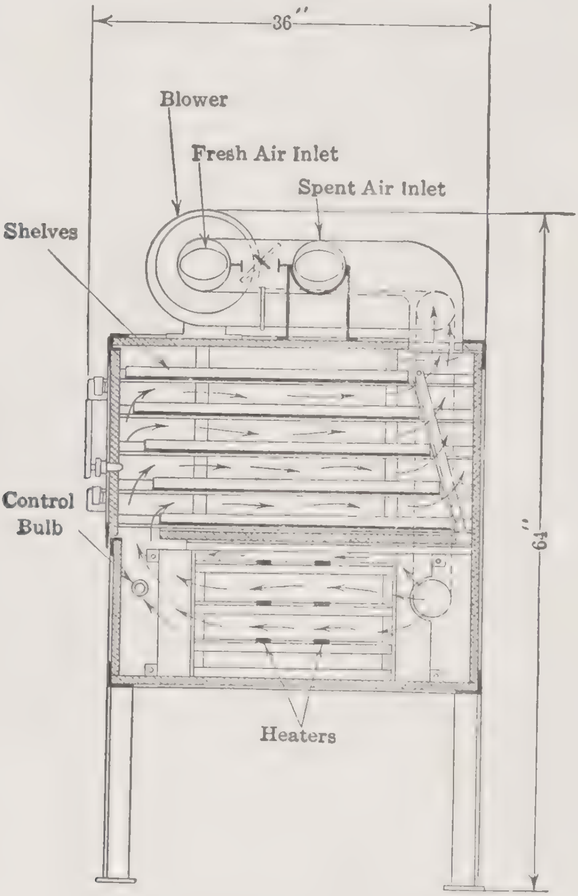


Fig. 7.—Stokes Electric Tray Drier.

A Keuffel and Esser 6 inch anemometer is satisfactory for direct reading of air flow.

Provisions should be made for recirculating air and for varying the fraction of fresh air supplied.

Materials Used in Drying Experiments. Materials which are commonly used for experimental work in unit operation laboratories include sawdust, clay, and precipitated calcium carbonate paste for drying in trays, and sheets of wallboard for drying in sheet form. The drying on trays of soap bars and chips, clay, and of blocks of wood offer special

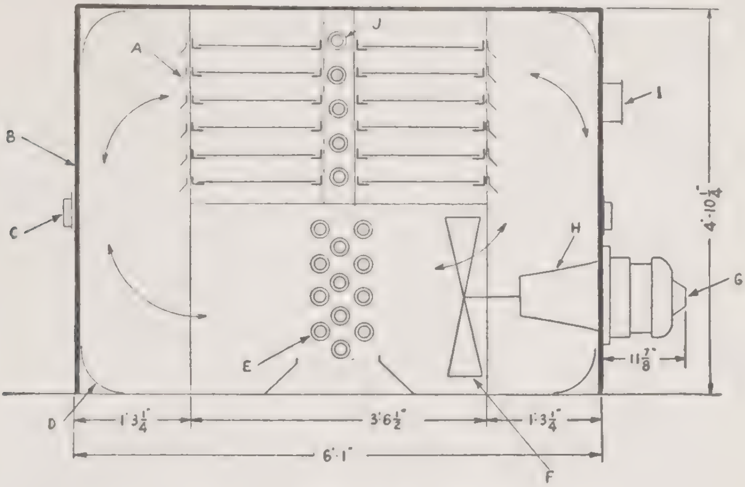


Fig. 8.—Cross-section of Proctor Standard Twelve Tray Drier.

A—Adjustable baffles assure uniform drying on all trays.
B—Insulation panel made of heavy sheet steel, inclosing 1½″ high efficiency insulation. Edges sealed with resilient insulating material.
C—Fresh air inlet and exhaust for moist air.
D—Curved interior.
E—Main heating coil, fin type.
F—High speed fan, special metal.
G—Motor and reversing mechanism.
H—Extended shaft motor. Fan mounted on motor shaft.
I—Reversing mechanism automatically alternates direction of air over trays.
J—Booster coils, fin type.

experiments where movement of moisture through the solid is slow compared to surface evaporation.

EXPERIMENT NO. 31

Equilibrium Moisture Content

Object. To determine equilibrium moisture contents of a sample by desorption and absorption.

As previously stated two different values of equilibrium moisture content may be obtained depending upon whether equilibrium is approached by sorption or desorption. The desorption value is higher than the other, and is the one of interest in drying. Desorption values of a sheet material such as textiles, yarns, cloth, or leather, can be obtained

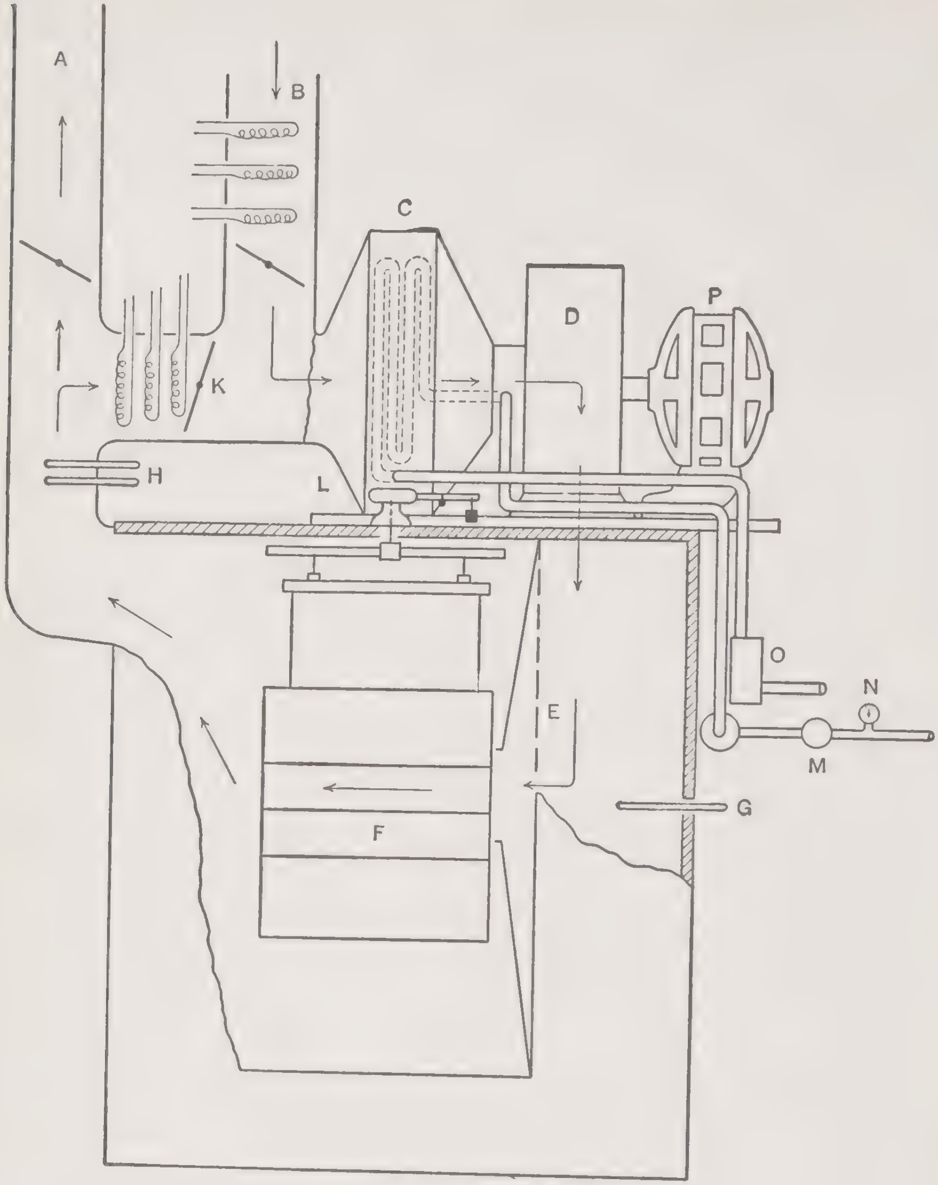


FIG. 9.—Experimental Drier.

A—Air Outlet.
B—Air Inlet.
C—Heater.
D—Fan.
E—Air Regulation Shutters.
F—Pan.
G—Inlet Wet and Dry Bulb Thermometers.

H—Outlet Thermometers.
I–J—Thomas Meters.
K—By-pass Damper for Recirculation.
L—Scale.
M—Temperature Regulator.
N—Steam Cage.
O—Trap.
P—Motor.

by starting with a wetted sample and suspending in an atmosphere of known temperature and humidity until a constant weight has been attained. The samples should then be suspended in a drying oven at 105° C. for two hours or until a nearly constant weight is obtained and the weight of dry sample recorded. The desorption equilibrium value of moisture content is expressed on the basis of the dry sample. Samples to be weighed after oven drying must be cooled in a closed, air tight weighing bottle before weighing.

The time required for conditioning of the sample depends upon the size of sample, temperature and circulation of air. Textile fabrics require 24 hours to attain equilibrium when freely exposed to uncirculated air; leather samples may require 5 days. For conditioning tests the samples should be suspended in containers and kept sealed in a room or bath at constant temperature. The humidity of the air in the container can be controlled by solutions of chemicals in various concentrations. The volume of solution used should be sufficiently large so that its concentration will not be significantly affected by the drying of the sample. For precision it is advisable to measure the concentration of the solution at the end of the test. Solutions of sulphuric acid in various concentrations have been successfully used to control relative humidity.

TABLE 1

RELATIVE HUMIDITY

	Percentage H_2SO_4	
	At 80° F.	At 120° F.
10%	64.8	66.6
25%	55.9	57.5
35%	50.9	52.5
50%	45.4	44.8
65%	36.0	37.1
75%	30.4	31.4
90%	18.5	19.2

Typical desorption equilibrium values are shown in Fig. 4 for chrome leather.

A better procedure for measuring equilibrium moisture contents is to employ a special laboratory drying apparatus for precision control in drying small samples. Such a drier is built by the Carrier Engineering Corporation. This drier provides for circulation of air at controlled temperature and humidity.

EXPERIMENT NO. 32

AIR DRYING OF A GRANULAR SOLID IN TRAYS

Object. To determine the rate of drying of sand under conditions of constant entering air temperature and constant air flow in an air cabinet drier. It is also required that the drying coefficient of the sand be determined during the various stages of drying.

Procedure. Inspect the drier and sketch its essential features showing the method of heating the air, the path of air travel, the method of temperature control of steam supply.

Do not start drier without the instructor's attention.

Before starting, the following precautions should be taken:

Make sure the air pressure for recorder control will be on for the entire period of run.

Open wide the drain valve on the steam line.

Open the main steam valve slowly until all the condensate and rust are blown out.

Close the drain valve until only a slight amount of steam leaves through it.

Open wide the steam valves on the heating coils.

Drain the steam pressure gauge of condensate.

Open slightly the condensate drip valve on the bottom of the heating coil.

Set the reducing valve to a low pressure to minimize the fluctuations in temperature.

Start blower with motor starting box.

Adjust air intake and outlet damper.

Start blower and drain moisture trap.

Adjust the temperature controller for desired temperature.

Adjust instruments for recording temperature and humidity where such are employed.

Allow the drier to operate with shelves empty in order to arrive at a steady state before inserting stock.

Take wet and dry bulb temperature readings of entering air before inserting stock to prove the absence of internal steam leaks.

A layer of dry sand is placed in a tray to a given thickness. The area of the pan should be measured. The rack holding the tray should be suspended from an overhead balance or platform scale and balanced against the weight of rack and empty pan so as to read directly the weight of contents. The weight of dry sand is recorded. The sand is saturated with water. After the drier has been brought up to a steady temperature, humidity and air flow the filled pans are inserted. The rate of air flow over the pan can be measured with a Prandtl Pitot tube or anemometer. The inlet and outlet wet and dry bulb thermometers are read at regular intervals. The rate of drying is measured by setting the overhead balance for some predetermined loss in weight and the time interval for attaining this loss measured by a stop watch. This operation is repeated for successive losses in weight.

It is required to determine the drying rates during all stages of drying sand.

SAMPLE DATA AND CALCULATIONS

The results obtained with a drier such as shown in Fig. 9 will be presented for illustration. The drying rate for each small decrement of loss of water was taken as representing the value for the arithmetic mean value of water content. The balance used had a special scale which later was converted to grams.

Average air temperature	75.8° C.
Average wet bulb temperature	35.9° C.
Weight of dry sand	2460 grams
Size of sand # 1/0, average	0.005 cm. radius
Thickness of layer	2.54 cm.
Area of pan	770 sq. cm.

The balance recorded the weight of wet sand only, having been previously balanced for weight of rack and empty pan. To convert scale readings to grams multiply by factor 456. A tabulation of experimental and calculated values is shown in Table 1. The method of making calculations is obvious from the tabulation.

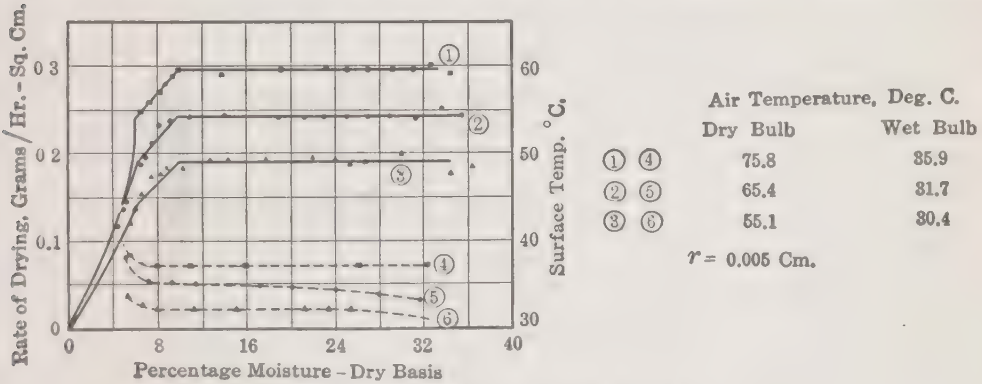


FIG. 10.—Rate of Drying Sand.[6]

Temperatures are given in dashed lines.

The drying rates are shown recorded in curve (1) of Fig. 10. It will be observed that three distinct stages of drying are manifest.

Experiment 32 can also conveniently be performed with sawdust to illustrate consideration of the presence of equilibrium moisture content under constant drying conditions.

EXPERIMENT NO. 33

Drying a Sheet Material with Recirculation of Air

Object. To obtain data on drying by recirculation of air.

Frequently it has been desirable to obtain experimental data under the variable drying conditions obtained in a progressive drier, where the stock travels counter to a steady stream of air but is exposed as it advances to air of constantly decreasing humidity. Progressive driers are usually not available nor practicable for experimental purposes, but the same data can be obtained in a cabinet drier by recirculation of a fraction of the air, starting with saturated air and allowing the unsaturation to decrease as drying proceeds. A constant temperature of entering air is desired. A wall board is selected for this experiment, since it represents a cheap fibrous material illustrating the effect of equilibrium moisture content. Wall board is particularly appropriate for tests because it is rigid enough to be dried in a horizontal as well as vertical position.

Procedure. Obtain from instructor a number of sheets of wall board corresponding to the number of shelves and cut to fit the size of the shelves. These should have been previously cut and soaked in water to complete saturation. Each sheet should be numbered.

Where the drier is provided with a platform scale on top, for direct weighing of stock while in the drier, weighings should be made at regular time intervals, for example, each ten minutes. Direct weighing while in drier is always preferred to avoid interruptions in opening drier.

Where variations in drying rates at different parts of drier are being investigated, it will be necessary to remove sheets and weigh individually, keeping the blower running continuously. The separate sheets should be weighed immediately and returned to the drier as soon as possible. Care should be taken to keep the door of the drier open as briefly and as little as possible.

The humidity of the air supply to the drier shall be determined by a sling psychrometer used in the immediate neighborhood of the drier. The humidity of the air entering and leaving the wall board shall be measured by wet and dry bulb thermometers placed in the air stream. If the velocity of the air stream passing over thermometers is sufficiently high, correct temperatures will be recorded by the actual readings. In

TABLE 2

Calculated and Experimental Data on the Drying of Sand

Experimental		Calculated		Experimental	Calculated				
Predetermined Setting of Balance	Corresponding Weight in Grams	Total Weight of Free Water in Grams	Total Loss of Water in Grams	Time Interval for Predetermined Loss of Water in Minutes	Time Interval in Hours	Rate of Drying for Entire Pan in Grams per Hour	Rate of Drying per Sq. Cm. in Grams per Hour	Water Content Grams per Gram Dry Sand	Average Water Content Over Time Interval, Gm. per Gm. Dry Sand
7.08	3233.1	873.1	.....	......	......	.....	.....	35.5	.....
6.98	3187.4	827.4	45.7	12.50	0.2085	218	0.283	33.6	34.45
6.87	3177.2	777.2	50.2	13.25	0.221	226	0.293	31.6	32.6
6.77	3091.5	731.5	45.7	12.166	0.203	225	0.291	29.7	30.66
6.67	3045.8	685.8	45.7	12.166	0.203	225	0.291	27.8	28.75
6.57	3000.1	640.1	45.7	12.166	0.203	225	0.291	26.0	26.7
6.47	2954.4	594.4	45.7	12.25	0.2045	222.5	0.288	24.1	25.05
6.37	2908.7	548.7	45.7	12.083	0.202	225.5	0.292	22.2	23.15
6.07	2771.6	411.6	137.1	36.583	0.609	225	0.291	16.74	19.47
5.75	2625.3	265.3	146.3	40.00	0.666	219	0.284	10.75	13.75
5.71	2607.0	247.0	18.3	4.833	0.0805	225	0.291	10.00	10.37
5.67	2588.7	228.7	18.3	5.083	0.0848	215	0.279	9.28	9.64
5.63	2570.4	210.4	18.3	5.25	0.0875	208	0.270	8.56	8.91
5.59	2552.1	192.1	18.3	5.417	0.0905	202	0.262	7.79	8.16
5.55	2533.8	173.8	18.3	5.666	0.0944	194	0.251	7.05	7.42
5.51	2515.5	155.5	18.3	5.883	0.0973	187	0.243	6.31	6.68
5.47	2497.2	137.2	18.3	6.583	0.1098	166	0.217	5.57	5.96
5.43	2478.9	118.9	18.3	10.5	0.1752	106	0.138	4.83	5.20
....	2360.0	0							

some laboratories, special provision for rapid circulation of air over thermometers is provided by placing thermometers in wells and rapidly passing a portion of the main air stream through these wells by a water aspirator to insure correct readings. These thermometers should be arranged for external reading and records made at 10 minute intervals for the first two hours and at 30 minute intervals thereafter. Readings of air flow over stock at various positions may be measured with an anemometer at 15 minute intervals during the first two hours and at 30 minute intervals thereafter. The experiment shall run continuously for eight hours or until the sheets acquire practically constant weight.

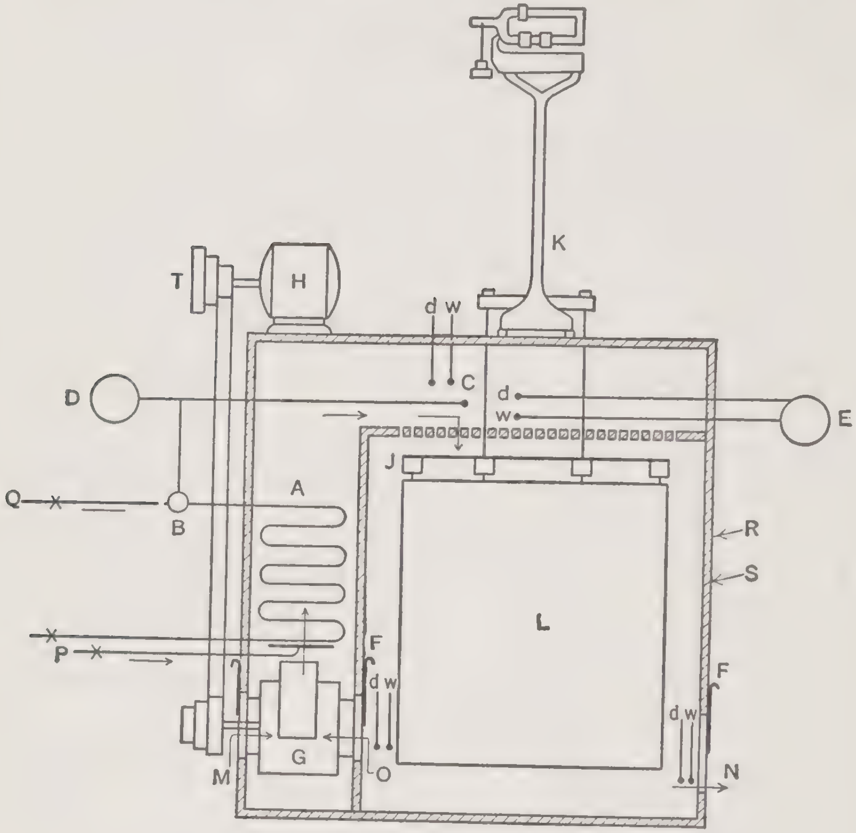


Fig. 11.—Experimental Drier for Vertical Sheets.

d—Dry bulb thermometers.
w—Wet bulb thermometers.
A—Heating coil.
B—Steam inlet automatic control valve.
C—Automatic temperature regulator.
D—Temperature recorder.
E—Humidity recorder.
F—Shutter for control of circulation.
G—Blower.
H—Motor.
J—Frame for supporting sheets.
K—Platform scale.
L—Vertical sheets.
M—Inlet for fresh air.
N—Outlet for spent air.
O—Opening for recirculated air.
P—Live steam supply.
Q—Steam supply to heater.
R—Insulation.
S—Sheet metal lined inside.
T—Adjustable pulley.

The rate of air flow may also be obtained from a water balance as previously described (page 257).

At the end of the drying experiment a sample of wall board should be taken to determine the final moisture content by drying to constant weight in a laboratory air oven at 105° C.

Heat Balance and Efficiencies. On the basis of the first hour of drying, a heat balance, drying efficiency, and pounds of water evaporated per pound of steam used should be calculated.

Calculate the thermal efficiency on the basis of the heat theoretically required to preheat the stock and to evaporate water compared to the heat given up by the steam supply. The weight of condensate and quality of steam supply must be known.

SAMPLE DATA AND CALCULATIONS

An example of the data taken and the treatment of these data will be made apparent by an illustration applied to the drying of chrome leather in vertical sheets. The methods of support and weighing in place are shown in Fig. 11.

Stock	30 calfskins
Average temperature of air entering stock	120.5° F.
Spacing of sheets	1 in. apart
Average thickness of sheets	0.0386 in.
Initial moisture content	1.48 lb. per lb. dry stock
Total weight of stock—initial	75.4 lb.
Total dry weight of stock	30.4 lb.
Average velocity of air over sheets	20.2 lb. per sq. ft./min.

A record of measurements made during the drying period is tabulated in Table 3, and shown graphically in Fig. 11. The calculated data are recorded in Table 4.

Calculations. The unsaturation of air entering the stock ΔH_1 and leaving the stock ΔH_2, are calculated from the dry and wet bulb readings from a humidity chart or from the relationship

$$\Delta H = 0.000238(t - t_w)$$

where t = dry bulb temperature in degrees Fahrenheit
t_w = wet bulb temperature in degrees Fahrenheit

This relationship can be used since the surfaces of the sheets were at the wet bulb temperature during drying. Since the change in unsaturation in passing the air over the sheets is small, the arithmetic mean difference may be taken at any instant,

$$\Delta H_m = \frac{\Delta H_1 + \Delta H_2}{2}$$

TABLE 3

Time, Minutes	Weight of Stock, Pounds	Weight of Water, Pounds	Temperatures, ° F.					
			Fresh Air		Air Entering		Air Leaving	
			Dry	Wet	Dry	Wet	Dry	Wet
0	75.4	45.0	....	....	124.2	118.5	123.35	117.6
5	74.6	44.2	....	....	121.2	114.4	120.45	114.25
10	73.5	43.1	....	....	119.0	112.0	118.25	112.05
15	72.7	42.3	....	....	118.5	110.4	117.3	110.8
20	71.8	41.4	....	....	120.0	111.3	118.1	111.5
25	70.9	40.5	....	....	121.5	112.0	118.7	111.8
30	69.9	39.5	....	....	120.1	109.5	118.7	109.4
40	67.5	37.1	....	....	120.1	109.0	118.7	109.1
50	64.3	33.9	89.0	70.0	121.3	104.9	119.5	106.4
60	61.3	30.9	87.5	73.0	121.0	103.9	120.3	104.5
70	57.7	27.3	....	....	121.6	103.2	119.9	103.7
80	55.3	24.9	87.8	72.0	121.5	102.1	120.5	102.9
90	52.7	22.3	89.0	72.4	121.8	101.1	120.1	101.6
100	50.0	19.6	....	....	121.2	99.6	119.7	100.2
120	45.5	15.1	87.0	70.2	119.0	96.1	118.1	97.0
130	43.5	13.1	....	....	120.2	95.2	118.5	96.0
140	42.1	11.7	89.2	70.8	120.8	94.0	118.7	95.0
150	40.5	10.1	....	....	120.4	92.5	118.3	93.6
160	39.4	9.0	89.0	70.4	120.3	91.0	118.3	92.3
170	37.9	7.6	....	....	120.0	89.8	118.5	91.0
180	36.9	6.5	....	....	120.2	89.0	118.5	90.1
190	36.0	5.6	....	....	120.7	87.2	118.6	88.4
200	35.4	5.0	87.5	67.4	118.0	84.8	116.9	86.4
210	34.9	4.5	....	....	119.1	84.0	117.9	86.0
220	34.4	4.0	....	....	119.9	83.4	118.2	85.5
230	34.0	3.6	....	....	119.3	82.3	117.6	84.5

The unsaturation of air increases progressively during drying. Since readings were taken at short time intervals the average unsaturation for any time interval ΔH_i may be be taken as the arithmetic mean of the values at the beginning and end of this period. The average value of unsaturation of air for the entire period elapsed can now be obtained by the graphical integration of ΔH_i plotted against time for the separate periods of total time elapsed.

$$\Delta H_{\text{ave}} = \frac{\int_0^\theta \Delta H_i d\theta}{\theta}$$

The equilibrium moisture content of the stock may be obtained from Fig. 4 for the required temperature and relative humidity. The equilibrium moisture content decreases as drying proceeds. The free moisture content, F, is then equal to $w - E$.

The rate of drying of the stock corresponded to the first falling rate period. The constant rate period did not appear, since excess water was initially scraped from the leather by hand setting.

From Equation (10)

$$\frac{dF}{d\theta} = - B_2 F \Delta H \tag{10}$$

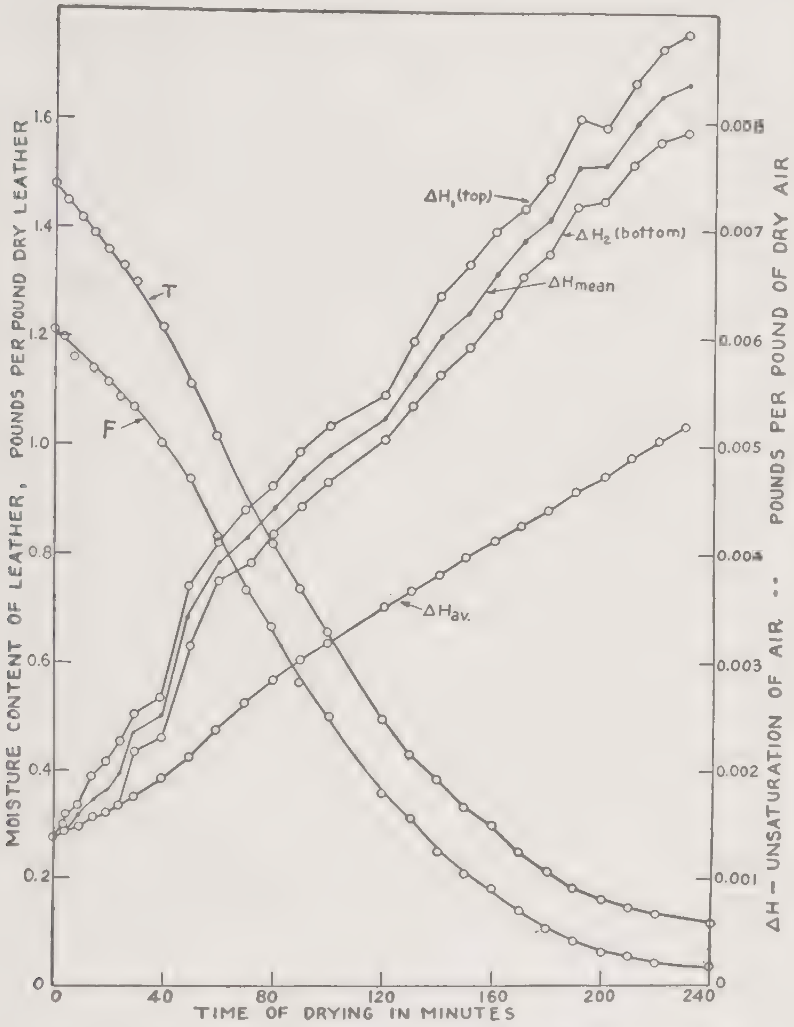


Fig. 12.—Direct Plot of Experimental Data of Experiment 33.

Since values of ΔH have already been integrated with respect to time, Equation (10) may be written

$$\frac{dF}{F} = - B_2 \Delta H_{av} d\theta \tag{11}$$

TABLE 4

Calculated Data of Experiment 33

Time θ Min.	Recirculated Air		Spent Air $t - t_w$ °F.	Unsaturation of Air $(H_w - H)10^3$					w	E	F	$\theta \Delta H_{av}$	Average in Drier		
	t °F.	$t - t_w$ °F.		Recirculated ΔH_1	Spent ΔH_2	Mean ΔH_m	ΔH_i for Time Interval	ΔH_{av} for Time Elapsed					t	$t - t_w$	Relative Humidity
				←Pound per Pound Dry Air→											
0	124.2	5.7	5.7	1.355	1.355	1.355	.....	.....	1.48	.27	1.21	0.	123.8	5.7	83
5	121.2	6.8	6.2	1.62	1.45	1.52	1.44	1.44	1.45	.255	1.195	7.2	120.8	6.6	80
10	119.0	7.0	6.2	1.665	1.475	1.57	1.545	1.49	1.418	.257	1.161	14.9	118.6	6.6	80
15	118.5	8.1	6.5	1.93	1.545	1.74	1.655	1.55	1.39	.25	1.14	23.2	117.9	7.3	79
20	120.0	8.7	6.6	2.07	1.57	1.82	1.78	1.605	1.36	.248	1.112	32.	119.0	7.65	78
25	121.5	9.5	6.9	2.26	1.64	1.95	1.885	1.66	1.33	.245	1.085	41.5	120.1	8.2	77
30	120.1	10.6	9.2	2.52	2.19	2.34	2.195	1.75	1.30	.23	1.07	52.5	119.4	9.9	72
40	120.1	11.1	9.6	2.64	2.28	2.46	2.40	1.91	1.22	.215	1.005	76.5	119.4	10.35	71
50	121.3	15.4	13.1	3.67	3.12	3.40	2.93	2.12	1.114	.178	.936	106.	120.4	14.3	62
60	121.0	17.1	15.7	4.07	3.74	3.905	3.65	2.37	1.015	.185	.830	142.	120.6	14.2	61
70	121.6	18.4	16.2	4.38	3.86	4.12	4.01	2.61	.897	.17	.727	182.5	120.8	17.3	56
80	121.5	19.4	17.6	4.62	4.19	4.405	4.26	2.81	.819	.158	.661	225.	121.0	18.5	52.5
90	121.8	20.7	18.5	4.93	4.41	4.67	4.54	3.01	.733	.155	.578	270.	121.0	19.6	50
100	121.2	21.6	19.5	5.14	4.64	4.89	4.78	3.18	.646	.150	.496	318.	120.4	20.6	48
120	119.0	22.9	21.1	5.45	5.03	5.24	5.065	3.50	.496	.14	.356	420.	118.5	22.0	44.5
130	120.2	25.0	22.5	5.95	5.35	5.65	5.445	3.64	.431	.13	.301	473.	119.4	23.7	41
140	120.8	26.8	23.7	6.37	5.64	6.005	5.83	3.80	.384	.13	.254	532.	119.8	25.2	39.5
150	120.4	27.9	24.7	6.65	5.88	6.265	6.135	3.96	.332	.122	.210	594.	119.4	26.3	37
160	120.3	29.3	26.1	6.96	6.21	6.58	6.42	4.11	.296	.115	.181	656.	119.3	27.7	35
170	120.0	30.2	27.5	7.19	6.55	6.87	6.725	4.26	.245	.11	.135	723.	119.2	28.9	32.5
180	120.2	31.2	28.4	7.43	6.75	7.095	6.98	4.42	.214	.105	.109	794.	119.3	29.8	30.5
190	120.7	33.5	30.2	7.98	7.20	7.59	7.34	4.57	.184	.10	.084	869.	119.6	31.8	28
200	118.0	33.2	30.5	7.90	7.25	7.555	7.57	4.72	.164	.10	.064	944.	117.5	31.8	27
210	119.1	35.1	31.9	8.35	7.60	7.98	7.77	4.87	.148	.092	.056	1020.	118.5	33.5	25
220	119.9	36.5	32.9	8.68	7.80	8.76	8.37	5.02	.132	.09	.042	1100.	119.0	34.7	25
230	119.3	37.0	33.1	8.80	7.88	8.34	8.55	5.17	.118	.085	.033	1190.	118.4	35.0	22

or upon integration

$$\ln \frac{F_0}{F} = B\Delta H_{\text{ave}}\theta \tag{12}$$

An average value of B for the entire run can be obtained by plotting log F against values of $\Delta H_{\text{av}}\Theta$ (Fig. 12); the points lie on a straight line. The slope of this line divided by 2.3 is equal to the drying coefficient, or $B = 3.11$.

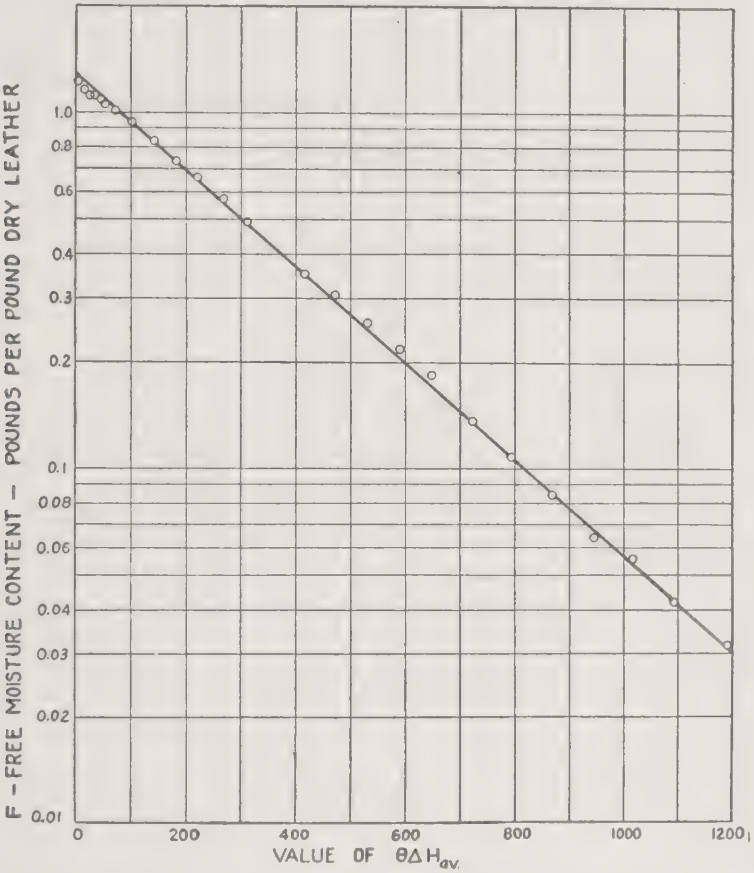


Fig. 13.—Evaluation of Drying Coefficient B (Experiment 33).

EXPERIMENT NO. 34

Effect of Air Velocity on the Drying of Wall Board

Repeat Experiment 33 at two other velocities during the constant rate and first falling rate periods. Plot the values of the drying coefficient against the log of the mass velocity of the air to obtain the relationship for the effect of velocity.

EXPERIMENT NO. 35

Effect of Thickness of Stock on Drying of Wall Board

Repeat Experiment 33 for two other thicknesses of the same wall board and determine the drying coefficient *B*. Determine the effect of stock thickness upon the coefficient *B* during the constant rate period and during the uniform falling rate period.

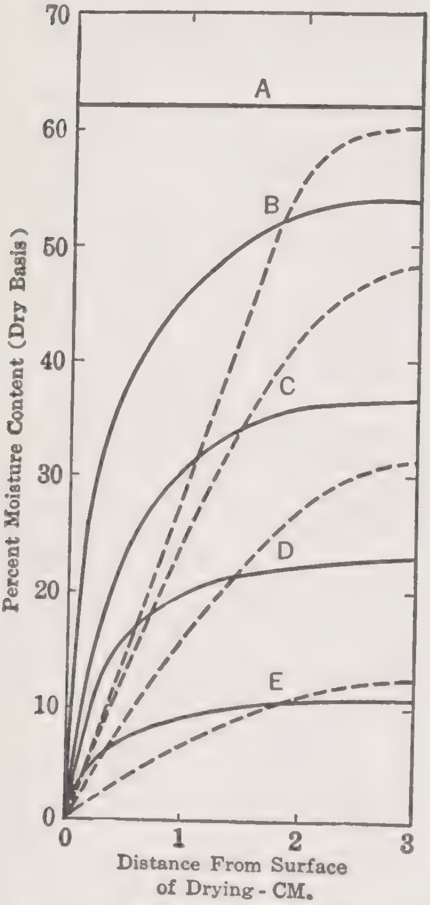


Fig. 14.—Moisture Distribution During the Drying of Soap.[14, 15]

Experimental ————

Calculated from Diffusion Equations — — — — —

EXPERIMENT NO. 36

Drying Colloidal and Fibrous Materials

Object. To study the drying of colloidal materials and also of hygroscopic fibrous solids in which the moisture content is below the fiber saturation point.

In fibrous materials, such as textiles, leather, wood, paper, and starch where the moisture content is below the fiber saturation point, the movement of water within the solid takes place by diffusion rather than by gravity, capillarity or external pressure. Also in colloidal materials, such as soaps, glues, gelatins, pastes, and fine clays, the movement of water occurs by diffusion. The rate of drying of such solids is relatively low and is controlled by the rate of diffusion of water through the solid, with evaporation taking place as rapidly as the water reaches the drying surface. Since the movement of water under such conditions is controlled by diffusion, the moisture distribution as well as the rate of drying can be calculated from the diffusion equations such as used for heat conduction in solids. In applying such equations to drying, average values of diffusivity may be employed to obtain the average moisture content and rate of drying at any instant but for obtaining moisture distribution the variable nature of diffusivity should be considered.[3] In contrast to heat conduction, drying proceeds with great structural changes taking place within the solid. In drying, marked changes in diffusivity take place as the moisture content and density of the solid changes; as shrinkage, checking, and cracking occur, and as cellular structure is modified. In Fig. 14 is shown the moisture distribution in the drying of soap compared with experimental values where a different constant value of diffusivity was assumed for each time of drying.[15] The results show that the diffusivity of moisture in soap rapidly falls off as the moisture content is reduced. Nevertheless average values of moisture content and rates of drying can often be calculated with fair agreement by using average values of diffusivities.

According to the diffusion equations:

$$\frac{dW'}{Ad\theta} = -\alpha \frac{dc}{dx} \tag{13}$$

$$\frac{\partial c}{\partial \theta} = \alpha \frac{\partial^2 c}{\partial x^2} \tag{14}$$

where W' = water flowing by diffusion
c = concentration of moisture in weight per unit volume at point x measured from the midplane in the direction of flow when the solid is drying from two opposite and parallel sides
α = diffusivity.

Equation (14) applies to conditions where diffusion is occurring in only one direction and requires the addition of other terms where drying is proceeding in radial or in more than one co-ordinate direction.[19]

Where diffusion occurs, the rate of water flow depends upon the first power of the moisture concentration gradient. With this assumption the concentration of moisture at any instant should vary inversely with the square of the distance from the center, and the average moisture content at any instant should vary inversely with the square of the thickness of the specimen.

In determining diffusivities, care must be taken to select specimens of uniform and symmetrical shape, and with uniform drying conditions prevailing on all exposed surfaces. To restrict drying to definite surfaces lead foil or paraffin may be used to seal the surfaces where drying is to be prevented. For example, the ends of short cylinders may be thus sealed where drying from the cylindrical surface only is desired.

Diffusivities can be calculated from the point moisture concentrations or from average moisture concentrations, each taken at frequent time intervals. By plotting the logarithm of unaccomplished moisture change of the solid against the ratio of time to the square of the radius, or half thickness, and comparing this with similar theoretical plots for the same shape the value of diffusivity can be calculated. Thus, for a specimen of given shape exposed to given drying conditions and for a given unaccomplished moisture change the diffusivity can be obtained from the following equation:

$$\alpha = \frac{Z}{\theta/R^2}$$

where Z = average unaccomplished moisture change in the solid

$= \frac{c - c_1}{c_0 - c_1}$

c = average moisture content, in weight per unit volume
c_0 = initial uniform moisture content
c_1 = moisture content in equilibrium with the air
R = half thickness of slab when drying from two sides
R = full thickness of slab when drying from one side
R = radius of cylinder when drying from cylindrical surface.

Values of Z may be obtained from Table IV which applies to the drying of solids by diffusion where the initial moisture content of the solid is constant, c_0, where the surface resistance to evaporation is negligible, and where the concentration of water at the surface remains in equilibrium with the moisture content of the air after drying starts. Under the same conditions where drying is taking place from more than one pair of surfaces as in bricks or short cylinders, the same values of Tables IV may still be used by combination of the Z values. Thus, for bricks $Z = (Z_a)(Z_b)(Z_c)$ where the subscripts refer to the three dimensions of the brick and

$$Z_{\text{cyl}} = Z_a Z_r$$

where a refers to the plane direction and r to the radial direction of the cylinder.

Procedure. (Taken from College of the City of New York).

Obtain six large bars of soap 3″ × 4″ × 13″. Shave 1/8 inch from each face of each cake. Cut all six cakes in halves, parallel to the largest faces, distinctly marking the halves of each cake.

Put one half of each cake aside for moisture determination. Cut three of the remaining 6 halves parallel to the largest face at one third of the thickness from one face. Thus there will be three groups of three cakes each having relative thickness of 1: 2: 3.

Cut 9 sheets of lead foil large enough to cover all the surface except one large face, the exterior face in the original cake of each of the nine prepared cakes. The foil should be applied with some pressure and

TABLE IV

Values of Z in Drying Solids, Assuming Diffusion Equations Apply[19]

$$E = \frac{c - c_1}{c_0 - c_1} = \frac{\text{average free moisture concentration at any time}}{\text{initial uniform free moisture concentration}}$$

$\frac{\alpha\theta}{R^2}$	Z_a	Z_r	$\frac{\alpha\theta}{R^2}$	Z_a	Z_r
	Slab	Cylinder		Slab	Cylinder
0.005	0.922	0.843	0.30	0.388	0.122
0.01	0.890	0.784	0.40	0.298	0.0684
0.02	0.839	0.698	0.50	0.236	0.0384
0.03	0.805	0.640	0.60	0.184	0.0215
0.04	0.773	0.588	0.70	0.144	0.0121
0.05	0.749	0.548	0.80	0.113	0.0068
0.06	0.725	0.512	0.90	0.088	0.0040
0.08	0.680	0.450	1.00	0.069	0.0021
0.10	0.643	0.394	1.50	0.020	0.0001
0.15	0.562	0.292	2.00	0.0058	0.0000
0.20	0.497	0.219	3.00	0.0005	
0.25	0.440	0.168			

evenly so that it will adhere uniformly and should just come up even with the edge of the uncovered face. Each cake of soap should be marked with its location on the shelf space. Each specimen should be weighed just before charging. When the drier has reached the conditions desired and automatically remains at those conditions for about 10 minutes, it should be charged. The process of drying is to be followed by weighing the specimens. For best results, about six or eight weighings, evenly spaced in time, should be made, about 45 to 90 minutes apart. The weighings must be made in the same order always, and rapidly, yet accurately. The drier doors should not be left open. If conditions are such that the drying is slow, it is preferable to take the weighings less frequently, and vice versa.

As soon as there is spare time, before or after starting the drying, representative moisture samples of the unused halves of the soap cakes should be removed by cork borers and retained in stoppered test tubes. The percentage of moisture is determined by the official method of the American Oil Association chemists, distillation with xylene in the Bidwell-Sterling apparatus.

Drying in Vacuum Chamber Driers

Where low temperature drying is required, below the normal boiling-point, where recovery of the liquid is desired or where the presence of air is objectionable, vacuum drying is resorted to. The maintenance of low pressures during drying requires the use of closed chambers operating intermittently. The chamber must be built to withstand a pressure of at least one atmosphere, or the full absolute pressure of the steam used in heating. The intermittent operation of a vacuum drier and the labor of opening and closing heavy doors sealed with bolts makes vacuum drying relatively expensive and limits its use to materials of high cost per pound, such as fine chemicals. The intermittent nature of operation permits unlimited flexibility as to the time allowed for drying.

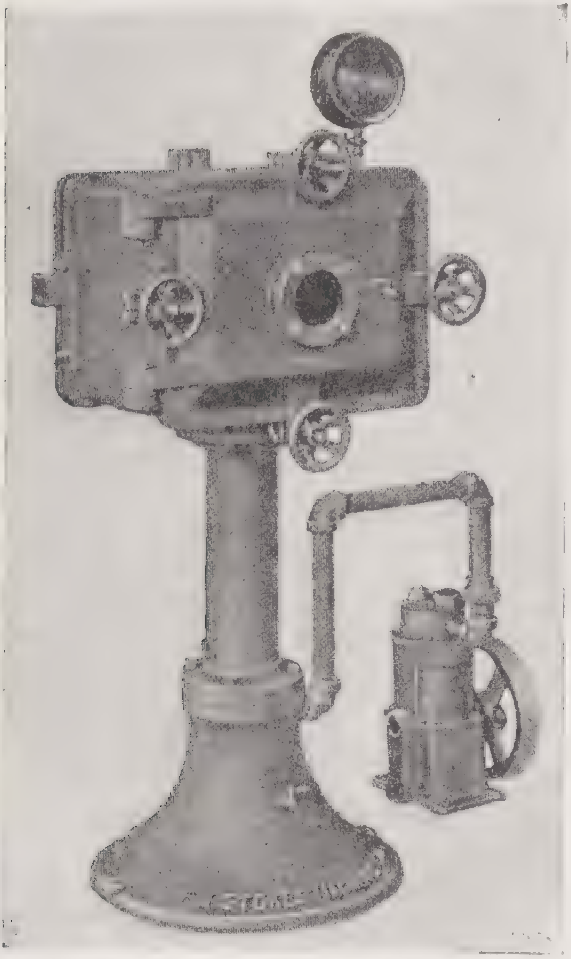


FIG. 15.—Stokes Vacuum Drier.

SPECIFICATIONS
Height—4′ 6″
Floor Space—2′ 3″ × 19″
Cu. Ft. Boxed—22
Net Weight—500 Lbs.
Gross Weight—625 Lbs.
Sq. Ft. Shelf Area—3
Sq. Ft. Cooling Surface—3

A popular vacuum compartment drier suitable for laboratory experiments consists of a horizontal shelf type with supporting frame (Fig. 15). A cross-section showing shelves and condenser is shown in Fig. 16. The shelves may be heated by pipe coils or channels carrying steam or hot water. The material to be dried is placed in pans fitting snugly upon the shelves. A vacuum is maintained by means of a surface condenser and rotary vacuum pump. The surface condenser should be provided with a receiver for the condensate. If steam is used for heating it is desirable to employ a reducing valve with an automatic temperature regulator on the steam supply.

Where hot water is used for heating it may be recirculated by a small centrifugal pump through a reservoir wherein the temperature is kept constant by the addition of steam or electric heat. The water velocity may be controlled by means of a valve located on the pump outlet, and may be measured by means of a sharp edge circular orifice inserted in a pipe union.

The drier should be provided with a vacuum gauge on the chamber and a pressure gauge on the steam line.

The advantage of vacuum drying is that it permits low temperature drying without the medium of air. Air drying is inefficient in that much of the heat supplied to the air is lost, despite recirculation. Recovery of heat from an air stream by condensation is not economical because of the slow rate of heat transfer in such condensers.

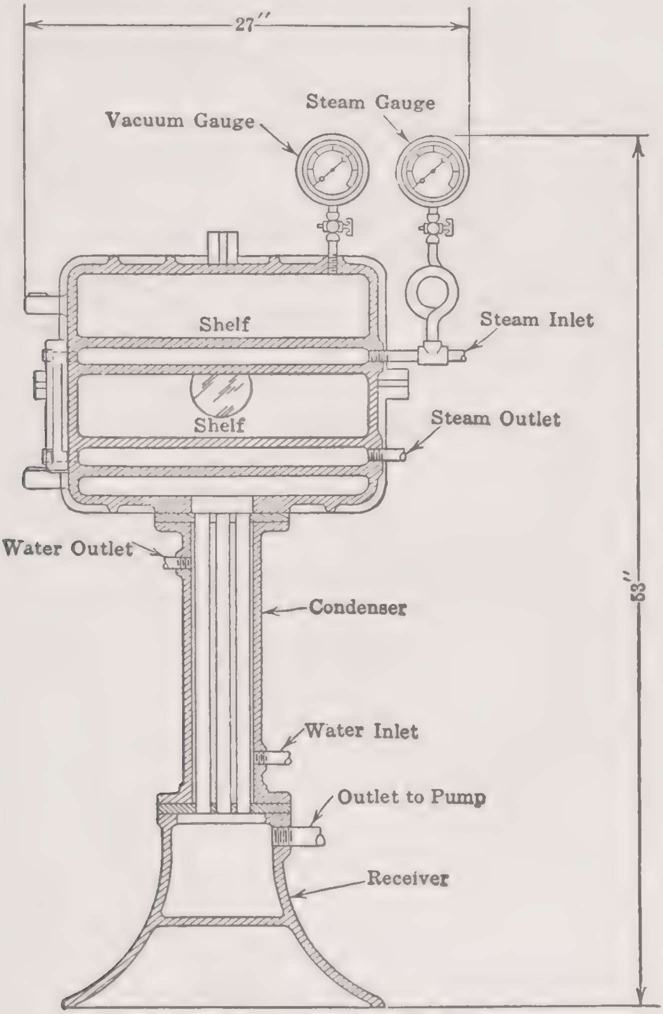


Fig. 16.—Stokes Vacuum Drier in Cross-section.

The heat required for evaporation is received by the stock by radiation from the shelf above and by transfer from the heating medium through the complex series of resistances encountered between the heating fluid and the stock. The hottest temperature appears at the bottom of the stock in contact with the shelf. Vaporization takes place at the bottom and is in turn condensed in the colder stock above. This transfer of heat tends to equalize the temperature throughout the stock. When the continuity of liquid film in the stock is broken by continued evaporation the free moisture eventually disappears completely, first at the bottom where the temperature is the hottest. As long as a continuous film is present the capillarity of the granular solid draws moisture to the driest surface and tends to keep the moisture content nearly uniform. During the last stage of drying, the heat must pass through progressively thicker layers of dry solid with corresponding decreasing values in overall heat transmission coefficients and drying rates. The change from a constant drying rate to a rapidly falling drying rate occurs suddenly. The corresponding average moisture content is called the critical moisture content. This value depends upon the nature of the stock and increases with its thickness. The critical concentration and the concentration gradients during vacuum drying are subjects meriting further theoretical and experimental investigation.

The presence of soluble substances elevates the boiling point and represses vapor pressure of the solvent and thus reduces the rate of evaporation. In drying fibrous materials, fine powders and colloidal substances the vapor pressure of the sorbed liquid is greatly repressed and drying will not continue below the equilibrium value. After the constant drying rate period the stock gradually increases in its average temperature, approaching the temperature of the shelves with a residual moisture content, if any, corresponding to the existing temperature and pressure.

The rate of evaporation depends upon the rate of heat transmission into the stock according to the general equation:

$$\frac{dQ}{Ad\theta} = K(t_m - t_s) \tag{15}$$

where $\frac{dQ}{Ad\theta}$ = rate of heat transfer per unit area

A = bottom area of stock in tray
t_m = average temperature of heating medium
t_s = average temperature of stock
K = the overall heat transmission coefficient as defined by the above equation
Q = heat transferred to the stock to evaporate water and also to preheat the wet stock and tray.

After the tray and wet stock reach a uniform temperature of evaporation, the heat transferred to the stock goes to the evaporation of water or

$$\frac{dQ}{Ad\theta} = \frac{\lambda_w dW}{Ad\theta} \tag{16}$$

where W = the liquid evaporated and
λ_w = the heat of vaporization of the liquid at the existing temperature which for the evaporation of free water corresponds to the existing pressure in the compartment.

Heat Balance. A complete heat balance of the vacuum drier during period of drying includes the following:

Heat Input. In case water is used for the heating fluid the heat input is equal to the water flowing times its drop in temperature. In case steam is used, the heat input is the enthalpy of the entering steam, allowing for quality or superheat, minus the enthalpy of the condensate.

Heat Output.

1. Heat used in preheating tray and dry stock from initial to final temperature.
2. Heat used in preheating water content from initial temperature to temperature of vaporization.
3. Heat used in vaporizing water (at temperature of vaporization).
4. Superheat in outgoing vapor (above temperature of condensation).
5. Radiation and convection losses from drier.

During a period of constant temperature

$\frac{dQ}{d\theta} = \frac{\lambda_w dW}{d\theta}$ and hence during this period combining Equations (15) and (16)

$$K = \frac{\lambda_w}{A(t_w - t_s)} \frac{dW}{d\theta} \tag{17}$$

The total resistance $1/K$ to the flow of heat is a complex structure made up of the separate resistances of the film of heating medium, scale and rust on surfaces, air space between shelf and tray, tray itself and stock. In addition heat is received by radiation from the shelf above the tray. The overall coefficient K is the sum of the coefficient of heat transfer due to radiation plus the reciprocal of the sum of all other resistances mentioned above. The resistance of the stock is small during the constant rate period, but becomes the major resistance in the last stages of drying of thick stock. To determine this coefficient K is one of the principal objects of these experiments. The temperature of the stock may be taken as the temperature corresponding to the boiling-point of pure water at the reduced pressure of the chamber. This will be true only for the removal of free moisture. The temperature will rise above this value for the bound water retained by fine capillary structures.

The thermal efficiency of a vacuum drier is arbitrarily taken as the ratio of the heat of vaporization of the liquid to the total heat input. Referring to the heat balance the

$$\text{thermal efficiency} = \frac{\text{item 3}}{\text{heat input}} \tag{18}$$

For the entire period of drying use the

$$\text{thermal efficiency} = \frac{\text{items 1, 2, 3 and 4}}{\text{heat input}} \tag{19}$$

This neglects the preheating of the pans and stock to the temperature of the chamber. The operation of a vacuum chamber is intermittent. Any attempt to ascertain a heat balance, heat transmission coefficients or thermal efficiency should be made during a period of drying, neglecting the periods of preheating and cooling the chamber or charge.

General Operating Instructions for Vacuum Chamber Driers:

Study the arrangement and piping of the drier to avoid mistakes in operation. Be sure the vacuum pump is well oiled during its operation, that water is running through the condenser, and that the condensate collector is drained before taking test samples.

Before heating the chamber, the steam trap should be opened to empty the heating shelves and trap from condensate. Before inserting trays for drying, the chamber should be preheated to constant temperature with door but slightly open. This will avoid unnecessary condensation of vapors on sides of chamber when drying starts. No material is to be placed in chamber until the gauge on the inlet steam line indicates the desired pressure. When the chamber is hot the steam trap waste line should be closed and the condensate collected. Make all arrangements for collection of condensate and weighings. Withdraw the condensed vapor occasionally, so as to avoid flooding of the collector chamber and damage to the vacuum pump.

In closing the door after charging it is necessary to tighten down on alternate sides of the door a little at a time to get a vacuum-tight seal. Do not strain the tightening lugs as the door is cast iron and may be easily broken.

After the charge is dry the valve to the air pump is to be closed and the air-cock opened.

When drying substances which are sensitive to heat, it is advisable to turn off the heating steam or hot water supply shortly before the termination of the drying-process since at that period over-heating of such substances may easily occur. Before starting have the instructor check over the equipment and preparation.

When experimental work is complete for the day, clean the apparatus and put in shape for future work.

EXPERIMENT NO. 37

TEST OF A VACUUM TRAY DRIER

This test and typical calculations were taken from the laboratory instructions of the Department of Chemical Engineering at the University of Minnesota.

Object. To determine:

1. The thermal efficiency of the drier during drying.
2. The drying rates of sand and sawdust.
3. The rates of heat transfer and drying on various shelves.
4. The values of the overall heat transfer coefficients with sand and sawdust.
5. Same as (4) with trays of Allegheny metal and enameled ware.

Equipment: *Devine* vacuum tray drier; four Allegheny metal trays; two enamel trays.

Procedure. A thermostatic regulator admits live steam to the water reservoir on the top of the drier, and a centrifugal pump circulates the hot water through the drier shelves. The water velocity may be controlled by means of a valve located on the pump outlet, and is measured by means of a sharp edge circular orifice inserted in a pipe union.

While the drier is being brought up to operating temperature, the material to be dried should be prepared, and the drying trays measured and weighed.

Four Allegheny metal trays and two enamel trays will be employed. These should be numbered with chalk or marking crayon upon an outside edge, and their weights and dimensions recorded.

Weigh out 1.5 kg. of sawdust in a tared dishpan. Place it in a 15 gallon crock and mix with water added in small portions until it is thoroughly damp, but not so wet as to lose water by drainage. Place the wet sawdust upon two Allegheny metal trays and one enamel tray, spreading it out evenly so as to cover the whole surface. Weigh each tray after filling.

Weigh out 15 kg. of sand and moisten in the same manner. Spread the wet sand upon the remaining trays and weigh. We may assume that the distribution of moisture is uniform in both sand and sawdust, thus enabling the calculation of the weight of bone dry material and water initially present on each tray.

Place the weighed trays in the drier in the following order:

1. Two Allegheny metal trays (one sand and one sawdust) on the top shelf. Here they will receive heat only by conduction from the shelf upon which they rest.

2. The second pair of Allegheny metal trays on the second shelf from the top. These trays will receive heat by radiation from the shelf above as well as by conduction from the shelf beneath.

3. One of the enamel trays should be placed on each of the remaining shelves.

Close the drier door, and start the vacuum pump, recording the starting time. Oil cups on the pump should have been previously filled and adjusted to feed properly; cooling water to pump and condenser should have been turned on and the condenser receiver should have been drained. The centrifugal pump circulating hot water should have been in operation for some time in order to bring the drier up to temperature. The thermometer wells for measuring inlet and outlet water temperatures should have been filled with water, and the rate of water circulation should be adjusted to a constant value, to be maintained thereafter throughout the test.

After drying for 15 minutes, stop the vacuum pump and admit air to the drier. Remove the trays, weigh them quickly, and return them to their former positions. Close the door, start the vacuum pump and continue drying for another 15 minute period, counting drying time as that time during which the trays are under vacuum. The test may be discontinued when 6 to 8 drying periods have been completed. When the drier is loaded with a uniform charge in testing the drier as a whole, the weight of liquid evaporated may be measured from the condensate collected. This procedure avoids the uncertainties introduced due to interruption in opening and closing the drier.

REPORT

1. Average temperatures during each 15 minute period of inlet and outlet heating water.
2. Average reading (cm. of mercury counterbalanced by water) of flow meter during each period. This`should be maintained constant as closely as possible.
3. Average vacuum in drier during each period (inches of mercury).
4. Weights of the 6 individual trays empty, filled with wet material previous to first test, and at end of each drying period.
5. Weight of dry material originally taken.
6. Barometric reading.
7. Dimensions of trays.

The data keepers should minimize the bulk of data to be given to each student by having someone average the readings of flow meter, vacuum gauge, and heating water temperatures secured in each period, during the following period.

The experimental data and treatment of data as reported by N. R. Amundson, a student at Minnesota, are given below:

SAMPLE DATA AND CALCULATIONS

Weight of dry sand = 15 kg.
Weight of dry sawdust = 1.5 kg.
Barometric pressure (corrected) = 738.9 mm. Hg = 29.1 Hg

Dimensions of trays:

Allegheny metal..............	25.5 in. × 15 in. = 2.66 sq. ft.
Enameled iron................	30.5 " × 25.5 " = 5.4 " "

Manometer reading on water circulated = 5.3 cm. Hg against water at 30° C.

Orifice, $\frac{1}{2}$ in. diameter or 0.001365 sq. ft.
Pipe, $1\frac{1}{4}$ in. diameter or 0.01038 sq. ft.

Original water content of sand = 9.1%

Period	Water Temp.		Vacuum in Drier in Hg.	Gross Weight at End of 15-Min. Period in Grams					
	Inlet ° C.	Outlet ° C.		Tray 1	Tray 2	Tray 3	Tray 4	Tray 5	Tray 6
0	.....	.....	....	6863	5861	3352	3554	13,207	7000
1	67.82	66.22	21.7	6820	5820	3308	3540	13,047	6902
2	66.88	65.39	21.8	6754	5770	3257	3509	12,937	6792
3	68.33	66.41	21.7	6693	5715	3196	3470	12,800	6675
4	68.59	67.19	21.5	6630	5671	3146	3440	12,676	6570
5	68.19	66.79	21.4	6575	5641	3091	3411	12,590	6471
6	67.85	66.52	21.2	6551	5617	3058	3381	12,539	6370
Ave.	67.94	66.42	21.55						
Tare weight of pans............				2280	2265	2368	2346	4922	4966
Composition of pans...........				Allegheny metal				Enamel ware	
Material dried..................				Sand	Sand	Sawdust		Sand	Sawdust
Shelf..........................				2	1 (top)	2	(top) 1	3	4 Bottom

Calculation of amount of water circulated during each period:

The flow meter manometer tubes are located on either side of a one-half inch sharp edged orifice drilled through a circular piece of sheet iron which has been inserted in a pipe union on the pump suction side. Rubber gaskets on either side of the orifice plate prevent leaks. The orifice has been calibrated and has a coefficient of 0.74.

Let u_a = velocity of water in pipe in ft. per sec
u_o = velocity of water in orifice in ft. per sec

Since this orifice has no enlargement chamber the velocity of approach u_a is not negligible. It may be expressed in terms of u_o by the ratio of areas, thus:

$$\frac{u_a}{u_o} = \frac{0.001365}{0.01038} = 0.1315$$

$$u_a = 0.1315u_o$$

$$u_a^2 = 0.01732u_o^2$$

The velocity of flow can now be obtained from the equation:

$$\sqrt{u_o^2 - u_a^2} = c_o\sqrt{2g\Delta H} \tag{20}$$

The value of ΔH should be expressed in head of fluid flowing, in feet, and at the temperature of flow. The pressure drop across the orifice is read in centimeters of mercury balanced by water.

At 67° C. the density of water is 61.1 pounds/cu. ft.

Density of mercury in manometer at 30° C. is 13.52 relative to water at 67° C.

Therefore $$\Delta H = \frac{(5.3)(13.52 - 1.)}{(2.54)(12)} = 2.175$$

Substituting in Equation (20):

$$u_o = 0.74\sqrt{\frac{(64.4)(2.175)}{1 - 0.01732}} = 8.84 \text{ ft. per second}$$

The total weight of water Q delivered in 1½ hrs. is then

$$Q = (8.84)(1.5)(3600)(0.001365)(61.1) = 3975 \text{ pounds}$$

Thermal efficiency of the drier:

Total water evaporated = 2313 grams or 5.08 pounds

Barometric pressure = 29.1 in. Hg
Vacuum in drier = 21.55 in. Hg
Pressure in drier = 7.55 in. Hg or 3.71 lb. per sq. in.

The corresponding latent heat of vaporization is 1008 B.t.u. per lb.
Heat supplied in vaporizing water = 5.08 × 1008 = 5121 B.t.u.
Average inlet temperature of water = 67.94° C.
Average outlet temperature of water = 66.42
Temperature fall in water 1.51° C.
Heat supplied by water = 3975 × 1.52 × 1.8 = 10,900 B.t.u.
From Equation (18)

$$\text{thermal efficiency} = \frac{5121}{10{,}900} \times 100 = 47\%$$

Properly, Equation (19) should be used since the test was conducted over a period including the preheating of tray and wet stock. Items 1, 2 and 4 were considered negligible.

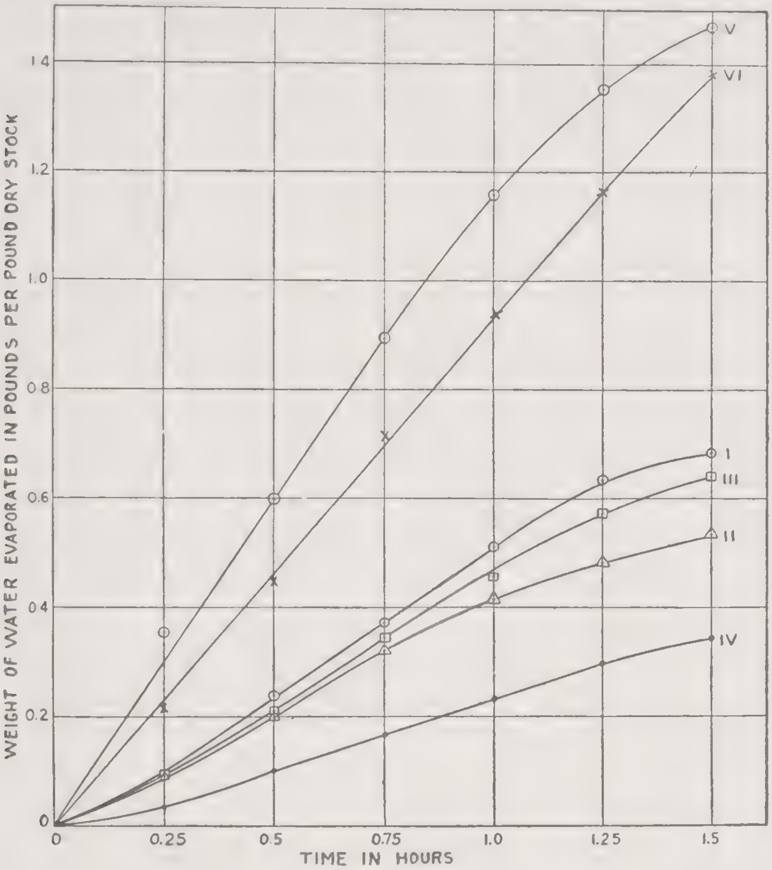


Fig. 17.—Water Evaporated. (Experiment 37.)

Tray	*Material*	*Shelf*
I	Sand	2
II	Sand	1 (top)
III	Sawdust	2
IV	Sawdust	1 (top)
V	Sand	3
VI	Sawdust	4 (bottom)

Drying Curves.

The water lost during each period and the cumulative loss from zero time were calculated for each tray. The curves for all six trays are plotted in Figure 17.

Gross weight before drying (tray 1) = 6863 grams
Gross weight after first 15 minutes = 6820 grams
Weight of water lost = 43 grams or 0.095 pound
Gross weight after second 15 minutes = 6754
Weight lost in 30 minutes = 109 or 0.240 pound

Original water content of sand, 9.1%.

or $$\frac{0.091}{1 - .091} = 0.10 \text{ pound of dry sand}$$

The drying rate per unit area is obtained by measuring the slope of curves in Figure 17 at the various time intervals and dividing by the tray area. The drying rates of sand and sawdust dried in Allegheny metal trays on shelf 2 were plotted against the free moisture content expressed in pounds per pound of dry solid. (Figure 18.)

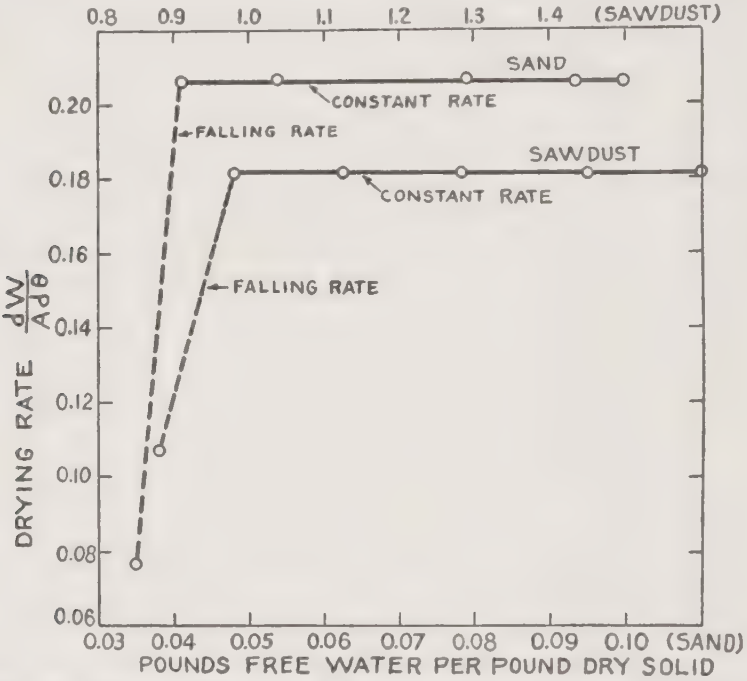


FIG. 18.—Rate of Drying in a Vacuum Drier. (Experiment 37.)

At the end of the first period with Tray 1 the tangent to curve is 0.549 and the area of tray is 2.66 sq. ft.

Hence $$\frac{dW}{Ad\theta} = \frac{0.549}{2.66} = 0.206$$

Calculated Data:

Cumulative water lost at end of each period in pounds:

Period	Tray 1	Tray 2	Tray 3	Tray 4	Tray 5	Tray 6
1	0.095	0.090	0.097	0.031	0.352	0.216
2	.240	.200	.209	.100	.594	.458
3	.374	.321	.343	.164	.896	.714
4	.512	.418	.453	.230	1.170	.946
5	.633	.484	.574	.294	1.359	1.164
6	.686	.537	.647	.343	1.471	1.386

Total water lost = 5.08 lb.

Pounds of free water at end of each period per pound of dry solid:

Period	Tray 1	Tray 2	Tray 3	Tray 4	Tray 5	Tray 6
1	0.0995	0.0981	1.695	1.79	0.0900	1.69
2	.0835	.0826	1.55	1.715	.0750	1.535
3	.0690	.0657	1.375	1.65	.0565	1.375
4	.0536	.0521	1.23	1.58	.0395	1.23
5	.0403	.0430	1.075	1.51	.0278	1.092
6	.0346	.0355	.980	1.46	.0210	.952
Dry solid......	9.1	7.14	0.769	0.944	16.19	1.59
Total free water	1.0	0.79	1.4	1.72	1.81	2.90

Drying rates—Pounds/hr.—sq. ft.

Period	Tray 1	Tray 2	Tray 3	Tray 4	Tray 5	Tray 6
1	0.188	0.163	0.158	0.088	0.206	0.175
2	.206	.188	.182	.098	.206	.175
3	.206	.188	.182	.098	.206	.175
4	.206	.150	.182	.098	.206	.175
5	.206	.126	.182	.098	.145	.175
6	.077	.119	.107	.075	.083	.175

Overall Heat Transmission Coefficients

The value of the overall heat transmission coefficient is calculated from Equation (17)

For example, for Tray 1

$$K = \frac{1008}{(2.66)(153 - 150)} (0.549) = 69.2 \text{ B.t.u./hr./sq. ft./}^\circ\text{ F.}$$

Summary of overall heat transmission constants during the constant rate period:

Tray	Shelf	Tray Material	Stock	K
1	2	Allegheny metal	Sand	69.22
2	1 (top)	" "	"	63.17 (no heating by radiation)
3	2	" "	Sawdust	61.14
4	1 (top)	" "	"	32.93 (no heating by radiation)
5	3	Enameled	Sand	70.00
6	4	"	Sawdust	58.80

Discussion of Results

1. The lowest rate of drying and the lowest effective heat transmission, other conditions being the same, were obtained on the top shelf. The tray on the top shelf loses heat by radiation to the roof of drier whereas the tray on second shelf receives radiation from the shelf above. Any condensation of moisture on roof dropping back into top trays must be guarded against.

2. Because of the superior thermal conductivity of sand over sawdust the values of overall heat transmission coefficients are higher with the

sand than with the sawdust. For the same reason the advantage of receiving radiant heat is particularly noticeable with the sawdust.

3. No advantage in drying rate or overall heat transmission was observed in using Allegheny metal over enameled ware for trays. Poor contact of the tray with the shelf may offset any advantage due to higher thermal conductivity of tray material.

Modifications of Experiment No. 37

Several other types of materials have been found suitable for student experiments such as sand, starch, crushed firebrick, and calcium carbonate paste. Other liquids such as benzol have been substituted for water.

In drying calcium carbonate paste, samples may be extracted by a cork borer at stated intervals and analyzed for $CaCO_3$ content by titration with standard acid.

It is also instructive to compare the rate of drying in a vacuum drier with results obtained in an air drier for the same temperature drop and moisture content.

EXPERIMENT NO. 38

Effect of Solids upon Rate of Evaporation

Repeat Experiment 37, using pure water in trays instead of wet sawdust. Compare the rate of evaporation in the two cases and report the effect of sawdust on the rate of evaporation of water for the same temperature conditions. Repeat the experiment with wet sand. This experiment was contributed by Ohio State University.

EXPERIMENT NO. 39

Uniformity of Evaporation Surface

This interesting experiment is contributed by the Chemical Engineering Department of Washington University, St. Louis.

Object. To determine the heat distribution and the rate of vaporization from various parts of the heating surface in a vacuum chamber drier. This particular experiment should reveal the uniformity of steam circulation inside the shelves and the effect of radiation on different shelves.

Procedure. The methods of operation are the same as in Experiment 37. Charge the drier pans with a number of small iron containers in each of which has been placed a known weight of water. When all the pans have been prepared insert them quickly into the vacuum drier which has been previously heated for at least one-half hour. Operate the drier for 20 minutes under the assigned conditions.

At the termination of the run open the drier, note the evaporation per unit area at each position in the drier. Plot a map showing the rate of vaporization at various points within the drier.

Questions on Vacuum Drying

How is the efficiency of liquid recovery determined?

What is the effect of reduced pressure on the boiling-point of liquids?

What is the effect of solid matter on the rate of evaporation of liquids?

What factors would you consider as influencing the efficiency of liquid recovery?

What changes in operation or set-up would you suggest to increase the efficiency of recovery?

What materials should be dried in a vacuum chamber drier?

What do the different slopes in the drying-rate-free moisture content diagram indicate?

What are the advantages of vacuum drying over air drying?

What effect will the use of different tray materials have upon the rate of evaporation? Why is this effect slight?

Why is the evaporation rate from top shelf less than from the others?

Nomenclature on Drying

A = area of surface where evaporation is taking place
B = drying coefficient (Eq. 5); B' (Eq. 8); B_1 (Eq. 9), B_2 (Eq. 10)
c = moisture content per unit volume
D = weight of dry stock, pounds
E = equilibrium moisture content, pounds of water per pound of dry stock
F = free moisture content
G = rate of air flow, vapor free basis
h = heat transfer coefficient, B.t.u. per hr./sq. ft./°F.
H = humidity of air, pounds of water vapor per pound of vapor-free air
k = transfer coefficient for water vapor (Eq. 1)
k' = transfer coefficient for water vapor (Eq. 2)
K = overall heat transfer coefficient
L = distance; also thickness of stock
p = partial pressure of water vapor
Q = heat transferred
R = half thickness of slab, when drying from two sides

R = full thickness of slab, when drying from one side
R = radius of cylinder
R' = rate of drying, lbs. per hr./sq. ft.
r = radius of curvature
t = temperature, °F.
T = surface tension of water, dynes per sq. cm.
u = linear velocity, ft. per second
V = volume, cu. ft.
W = water evaporated
W' = water flowing by diffusion
w = average water content, pounds of water per pound of dry stock
Z = unaccomplished average moisture change in solid
α = diffusivity
ρ = density of dry stock
θ = time elapsed
λ = latent heat of vaporization of water

Subscripts

a = in air stream
a, b, c, r = directions of diffusion
s = at surface; at saturation
i = initial; interval
av = average
m = arithmetic mean
v = vapor
w = at interface; at wet bulb temperature

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Chapter VII

HUMIDIFICATION AND DEHUMIDIFICATION

O. A. Hougen[1]

The operations of humidification, dehumidification, and the cooling of water by air are special cases of the absorption and of the desorption of gases where water vapor is the gas transferred. Unusual emphasis is given to these special cases because of their extensive applications in the conditioning of air for manufacturing operations, in the air drying of solids, and in the air cooling of water in towers and spray ponds.

DEFINITIONS

Humidification is the process of adding water vapor to air or other gas and is accomplished by passing air against a spray of water in a spray chamber or water scrubber, or by introducing a jet of steam into the air. The humidification of air always accompanies the air drying of a solid. **Dehumidification** is the process of removing water vapor from air or other gas and is usually accomplished by cooling the air either by a surface condenser or by a spray of water colder than the dew point of the air.

The *humidity* of air is conveniently expressed in pounds of water vapor on the basis of one pound of vapor free air. In dealing with gases other than air it is also convenient to use the term *molal humidity* which expresses the pound mols of water vapor on the basis of one pound mol of vapor-free gas.

The **degree of saturation** of a gas with water vapor is referred to either as relative humidity or as percentage humidity. *Relative humidity* is the ratio of the partial pressure p_a of the water vapor actually present to the vapor pressure of water p_s at the same temperature, that is,

$$\text{Relative humidity} = \frac{p_a}{p_s} \times 100 \tag{1}$$

Percentage humidity on the other hand is the ratio in percentage of the actual weight H_a of water vapor in air expressed in pounds per pound of dry air to the weight H_s of water vapor in air per pound of dry air when saturated at the same temperature and total pressure.

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$$\text{Percentage humidity} = \frac{H_a}{H_s} \times 100 \tag{2}$$

The term relative humidity has two advantages over the term percentage humidity; namely, for any given partial pressure of water vapor the relative humidity is independent of the total pressure, and the equilibrium moisture content (see page 236) of a hygroscopic material is independent of total pressure when related to relative humidity. On the contrary, for a given partial pressure of vapor, percentage humidity depends upon the total pressure. Percentage humidity, however, has the advantage of expressing results directly as a ratio of weights rather than of partial pressures, and hence is directly related to the weight balance. For this reason, in engineering calculations, the term percentage humidity is preferred. From Dalton's law

$$\frac{\dfrac{H}{18}}{\dfrac{1}{29}} = \frac{p}{P - p}$$

or

$$H = \left(\frac{18}{29}\right)\left(\frac{p}{P - p}\right) \tag{3}$$

where p is the partial pressure of water vapor and
P is the total pressure.

The relationship between relative humidity and percentage humidity is hence given by the equation,

$$(\text{Relative humidity}) = \frac{P - p_a}{P - p_s}\,(\text{percentage humidity}) \tag{4}$$

Since p_a is always less than p_s the numerical value of percentage humidity is less than that of relative humidity. The difference between the two diminishes with decreasing temperature and increasing degrees of saturation.

The *dew point* of a gas is the temperature to which the gas must be cooled by removing heat at constant humidity to just start the condensation of water. The temperature of the air at the point where condensation starts is at the dew point but the entire body of air need not be cooled to this temperature to start condensation. The *wet bulb temperature* refers to the equilibrium temperature attained by a water surface when water is vaporizing into air under adiabatic conditions, that is, when the water is evaporating at a constant temperature and in so doing is receiving heat only from the sensible heat content of the air and when neither air nor water is receiving heat by conduction or radiation from any other source.

The term *humid heat* s refers to the heat capacity of air on the basis of one pound of dry air with its associated water vapor H or to the heat capacity of $(1 + H)$ pounds of air-water vapor mixture,

$$s = c_a + c_w H = 0.238 + 0.48H \tag{5}$$

where c_a is the specific heat of the dry air and
c_w is the specific heat of the water vapor at constant pressure.

So-called *humidity charts* have been constructed to show the relationships among humidity, degree of saturation, dew point, dry and wet bulb temperatures. The most common humidity chart is for air at a pressure of one atmosphere, with the humidity expressed in pounds per pound of dry air, and with the degree of saturation expressed in percentage humidity. The molal humidity chart is more general in that it applies to all diatomic gases admixed with water vapor.

By *adiabatic cooling of air* is meant the cooling of the air stream by evaporation of water from a water surface which is kept at constant temperature; the sensible heat lost by the air stream is compensated by an equal gain in latent heat due to the vaporization of water into the air stream; no heat is received by the air by conduction or radiation. The saturation temperature reached by the air in adiabatic cooling is called the *temperature of adiabatic cooling at saturation.* The wet bulb temperature is the temperature of adiabatic saturation at the water interface whereas in adiabatic cooling the temperature of saturation refers to the temperature at which the entire gas stream becomes saturated and not merely at the interface. For air-water vapor mixtures the temperature of adiabatic cooling and the wet bulb temperature are fortuitously nearly identical at low temperatures. The distinction between the two is given on page 285.

Changes in Temperature and Humidity During Countercurrent Operations. The changes in temperature and humidity in countercurrent flow of air and water in the operations of adiabatic humidification, dehumidification and in the cooling of water by air are shown graphically in Figures 1 to 6.

T is the temperature of water
H^* is the humidity of air in equilibrium with water at temperature T
t is the dry bulb temperature of air
t_w is the wet bulb temperature of air
t_d is the dew point temperature of air
H is the humidity of air
H_w is the humidity of air saturated at its wet bulb temperature.

Subscript 1 refers to properties of entering air and water.

Subscript 2 refers to properties of leaving air and water.

In adiabatic humidification the temperature of the water remains substantially constant and at the wet bulb temperature of the air and hence the humidity of the air in equilibrium with the water is the same as the humidity of air at its wet bulb temperature, $T = t_w$, $H^* = H_w$ and $T_1 = T_2$. Sensible heat is flowing from the air to the surface of the water where the heat transferred is converted to the vaporization of water at the constant temperature of the surface. The resistance of the air film only is involved in adiabatic humidification.

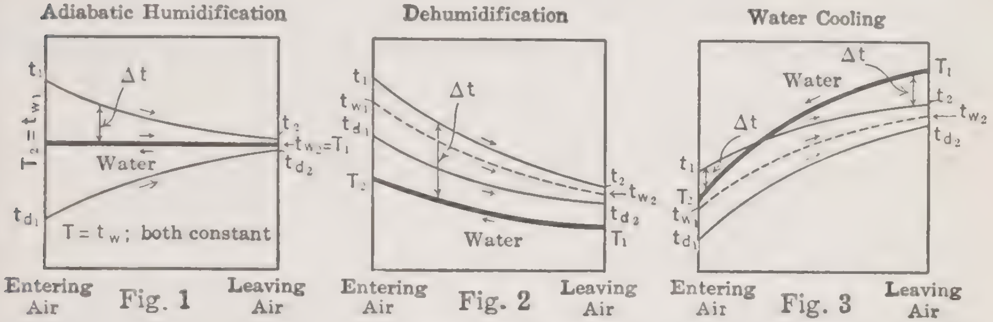

Fig. 1 Fig. 2 Fig. 3

Temperature Changes.

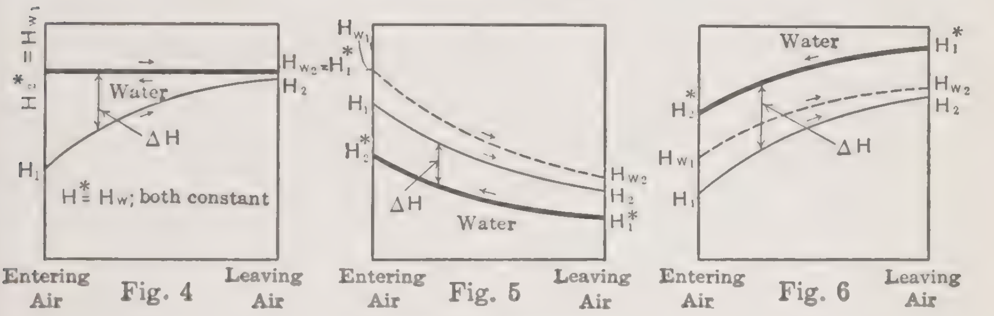

Fig. 4 Fig. 5 Fig. 6

Humidity Changes.

Figs. 1–6.—Temperature and Humidity Changes in Countercurrent Humidification, Dehumidification and Water Cooling.

In dehumidification the temperature of the water at any point is below the dew point of the air at the same point. The water gains heat by loss of sensible heat of the air and by condensation of water vapor. A temperature drop is established in both the air and water films at their common interface. The temperature of the interface is higher than the temperature of the main body of water and is also at or below the dew point of the air. In dehumidification the resistances of both air and water films are involved.

In cooling water by air in countercurrent flow, the water stream is losing heat both by vaporization and by flow of sensible heat to the colder air. At the bottom of the chamber the water may actually be cooled below the temperature of the air but not below its wet bulb temperature. In this latter situation the water is losing heat by evaporation but also receiving some by heat transfer from the warmer air. The humidity of air in equilibrium with the temperature of the water is always higher than the humidity of the air. In water cooling, the resistances of both air and water film are involved.

Heat Balances in Countercurrent Operations. Water in the liquid state at 32° F. is taken as the reference state. Let the following symbols apply:

W = rate of water flow.
T = temperature of water
t = temperature of air
G = rate of flow of dry air
c_a = specific heat of dry air
q = enthalpy of water vapor = $(t_{d1} - 32) + \lambda + c_w(t - t_d)$
t_d = dew point of air
c_w = specific heat of water vapor
λ = latent heat of vaporization of water at the dew point
$r = q + 32$.

Subscript 1 refers to properties of entering air and water.

Subscript 2 refers to properties of leaving air and water.

The following heat balance then applies to any of the operations of humidification, dehumidification or cooling of water by air.

Input	**Output**
1. In water entering $W_1(T_1 - 32)$	1. In water leaving $W_2(T_2 - 32)$
2. In dry air entering $G\,c_a(t_1 - 32)$	2. In dry air leaving $= G\,c_a(t_2 - 32)$
3. In water vapor entering $G\,H_1(r_1 - 32)$	3. In water vapor leaving $= G\,H_2(r_2 - 32)$
	4. Heat losses
Total Input = ...	Total Output = ...

In adiabatic humidification the heat losses or gains from the outside are zero. $W_1 - W_2$ is the water evaporated in humidification and water cooling, and $W_2 - W_1$ is the water condensed in dehumidification.

Theory of Humidification. When an air stream is humidified by passing it over a water surface in a humidifying chamber, the following weight balance results:

$$GdH = ka(p_s - p_a)dV \tag{6}$$

where G = rate of flow of dry air
H = humidity of the air
p_s = vapor pressure of the water at its surface temperature
p_a = partial pressure of the water vapor in the air stream
V = volume of the chamber
a = active interfacial area of water per unit volume
k = proportionality factor, or humidification coefficient as defined by Equation (6) with a numerical value dependent upon the units employed.

For any given spray chamber or scrubber, Equation (6) becomes:

$$G(H_2 - H_1) = kaV(\Delta p)_{av} \tag{7}$$

where H_2 = humidity of the air leaving
H_1 = humidity of the air entering
$(\Delta p)_{av}$ = average difference in driving potential of vapor pressure difference between the terminals of the chamber.

The value of the coefficient ka is influenced by all variables which change the thickness and properties of the air film, such as velocity of air flow and its temperature.

For adiabatic humidification the temperature of the water and hence its vapor pressure are constant. At low temperatures the mean value of $(\Delta p)_{av}$ is approximately equal to the logarithmic mean value;

$$(\Delta p)_{av} = \frac{\Delta p_2 - \Delta p_1}{\ln \dfrac{\Delta p_2}{\Delta p_1}} \tag{8}$$

where $\Delta p_1 = p_s - p_{a1}$ where air enters the humidifier
$\Delta p_2 = p_s - p_{a2}$ where air leaves the humidifier.

Instead of using a difference in partial pressures as a driving potential it is more convenient to use a difference in humidities since humidities are directly related to the weight balance of the process.

The driving potential expressed in humidities is $\Delta H = H_s - H$ where H_s is the humidity of the air when saturated at the surface temperature of the water.

Equation (3) can be approximated at a low temperature range and at one atmosphere pressure to

$$H = \frac{18}{29}p, \quad \text{hence } \Delta H = \frac{18}{29}(p_s - p) \tag{9}$$

location. It is not necessary to cool the entire air stream below the dew point to bring about condensation. For instance on a cold day frost will appear on the inside surface of a window pane of a heated building even though the air inside is far from saturated. When the air stream is passing through the spray water vapor will diffuse across the air film to the colder water and will there condense. The air stream will lose both sensible and latent heats and these will be transferred to the water spray. Both gas and liquid film resistances are encountered. The water spray will rise in temperature as dehumidification proceeds.

At any given point in the dehumidification

$$dq = h_1a(t - T)dv \tag{18}$$

and

$$GdH = k_1a(H - H^*)dV \tag{19}$$

where h_1a is the heat transmission coefficient in dehumidification and
k_1a is the mass transfer coefficient as defined by these equations
T is the temperature of the main body of the water spray
H^* is the equilibrium humidity of air corresponding to saturation at temperature T.

It will be observed from Figures 1 and 4, that both h_1a and k_1a are overall coefficients made up of the separate coefficients of both gas and liquid films. The overall coefficients can be calculated from the separate film coefficients but for convenience the composite values are employed.

Over the entire length of chamber Equations (18) and (19) may be written

$$q = h_1aV(\Delta t)_{\text{av}} \tag{20}$$

$$G(H_1 - H_2) = k_1aV(\Delta H)_{\text{av}} \tag{21}$$

The value of Δt at any point is the difference between the temperature of the air and the temperature of the main body of water (Fig. 2). This temperature drop includes the drop across both air and water films. Similarly ΔH is the humidity of air minus the humidity of air corresponding to saturation at the temperature of the main body of water; it includes the drop across the air film plus the equivalent drop across the water film. The average values of Δt and ΔH may be taken as the arithmetic mean where the values vary slightly through the chamber (page 289).

The temperature rise of the water can be calculated from the heat balance (page 288). Neglecting heat losses, this results in

$$T_2 - T_1 = \frac{G}{W_1}\left[c_a(t_1 - t_2) + (H_2 - H_1)(T_2 - 32) + H_1(q_1) - H_2(q_2)\right] \tag{22}$$

For practical purposes the heat balance can be simplified to the following:

$$T_2 - T_1 = \frac{G}{W_1}\left[\lambda_{av}\,(H_1 - H_2) + s_{av}\,(t_1 - t_2)\right] \tag{23}$$

The equation for the transfer of heat becomes

$$Gs_{av}\,(t_1 - t_2) = h_1 aV(t - T)_{av} \tag{24}$$

and for the transfer of water vapor,

$$G(H_1 - H_2) = k_1 aV(H - H^*)_{av} \tag{25}$$

Water Cooling by Air. Water from condensers and heat interchangers is frequently cooled by an air stream in spray ponds or in cooling towers employing natural draft or forced flow of the air. Water may be cooled by air as long as its temperature is above the wet bulb temperature of the air. If the flow of water and air are countercurrent, the water can be theoretically cooled to the wet bulb temperature of the entering air.

The general conditions of air and water streams in a water cooler are shown in Figs. 3 and 6. In water cooling, water is losing sensible heat by evaporation over the entire path but where the water is cooled countercurrently below the temperature of the entering air, the air will be cooler than the water at the top of the tower but warmer than the air at the bottom of the tower.

The heat balance of a water cooler may be obtained from page 288. For practical purposes a simplified heat balance is used, thus:

$$Gs_{av}(t_2 - t_1) + G(H_2 - H_1)\lambda_{av} = W_1(T_1 - T_2) \tag{26}$$

where s_{av} is average humid heat of the air stream and
λ_{av} is the average heat of vaporization of water in the cooler.

The water vaporized may be calculated from the diffusional process.

$$G(H_2 - H_1) = k_2 aV(H^* - H)_{av} \tag{27}$$

where k_2a is the overall coefficient for vaporization of water, including resistance of both water and air films

H^* is the equilibrium humidity of air corresponding to the temperature of the main body of water T.

For transfer of sensible heat

$$Gs_{av}(t_2 - t_1) = h_2 aV(T - t)_{av} \tag{28}$$

where h_2a is the overall heat transfer coefficient for flow of sensible heat from air to water.

are obtained. The air supply entering the spray tower should remain at constant temperature and humidity. For example, the air supply may be at 80° F. and 30% humidity, and be cooled adiabatically to 62° F. and 90% humidity, in which case the circulating water should be at 60.5° F. The air or water supply should be regulated so that complete saturation of the outgoing air is not obtained. Over a given time interval, calculate from the humidity chart the weight of water evaporated. From the weight of water evaporated per hour, the humidity difference in the tower ΔH_{av}, and the volume of the tower, calculate the humidification coefficient from Equation (11). From the rate of air flow and its change in temperature, calculate the heat transmission coefficient from Equation (13).

The effects of air velocity and rate of water flow on both coefficients may be studied subsequently as separate experiments.

Data on Adiabatic Humidification

Volume of packing or spray chamber = V cubic feet.
Diameter of tower = D feet.
Height of packing or spray chamber = L feet.
Rate of air flow = G pounds of dry air per minute.
Rate of water flow = W pounds of water per minute.

TEMPERATURES

Time	Water Entering	Water Leaving	Air Entering		Air Leaving	
			Dry Bulb	Wet Bulb	Dry Bulb	Wet Bulb
	T_1	T_2	t_1	t_{w_1}	t_2	t_{w_2}
....	....	....	....	....	....	....
....	....	....	....	....	....	....
....	....	....	....	....	....	....
....	....	....	....	....	....	....
Average...	....	....	....	....	....	....

HUMIDITIES

Time	Humidity of Entering Air			Humidity of Leaving Air				
	In Air	At Wet Bulb	ΔH Entering	In Air	At Wet Bulb	ΔH Leaving	ΔH_{av}	(H_2-H_1)
	H_1	H_{w_1}		H_2	H_{w_2}			
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
Average.	....	....	....	....	....	....	....	....

A heat balance is set up from Equation (13a).

The heat and mass transfer coefficients are calculated from Equations (11) and (13).

EXPERIMENT NO. 41

Dehumidification of Air

Where an air-conditioning plant is available, warm, humid air can be dehumidified by cooling the circulating spray water by means of a refrigerating unit. For experimental purposes equally instructive results may be obtained by passing the warm, humid air through a tower countercurrent to a spray of cold water. For example, the air may be heated in a separate chamber by injecting steam and maintained at a constant initial temperature and humidity by controlling the rate of air flow and steam injection. The water supply of the laboratory is often sufficiently cold and constant in temperature to serve the purpose of cooling the air and condensing part of the water vapor. The rates of air flow and water flow should be measured together with all the usual temperature and humidities. Establish both weight and heat balances. Calculate the water vapor and heat transmission coefficients from Equations (20), (21), and (23).

Volume of packing or spray chamber = V cubic feet.
Diameter of tower = D feet.
Height of packing or spray chamber = L feet.
Rate of air flow = G pounds of dry air per minute.
Rate of water flow = W pounds of water per minute.

HUMIDITIES

Time	Humidity of Air Entering			Humidity of Air Leaving				
	In Air	Corresponding to T_2	H_2	In Air	Corresponding to T_1	H_2	ΔH_{av}	H_1-H_2
	H_1	H_{2w}		H_2	H_{1w}			
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
....	....	....	....	....	....	....	....	....
Average.	....	....	....	....	....	....	....	....

A heat balance may be established from Equation (26) and heat and mass transfer coefficients calculated from Equations (27) and (28).

Nomenclature on Humidification and Dehumidification

a = active interfacial area per unit volume, sq. ft.
A = area of interchange surface, sq. ft.
A = area of cross-section of chamber
c_a = specific heat of air
c_w = specific heat of water vapor
D = diameter of tower, ft.
G = rate of flow of dry air
H = humidity of air, pounds of water vapor per pound of vapor free air
H^* = humidity of air, in equilibrium with water at temperature T
h = heat transmission coefficient of sensible heat
ha = heat transmission coefficient of sensible heat (Eq. 12)
h_1a = heat transmission coefficient of sensible heat (Eq. 18)
h_2a = heat transmission coefficient of sensible heat (Eq. 28)
k = humidification coefficient (Eq. 6)
k' = humidification coefficient (Eq. 11)
k_1a = dehumidification coefficient (Eq. 19)
k_2a = humidification coefficient (Eq. 27)
L = height of tower packing or chamber, ft.
p = partial pressure of water vapor
P = total pressure
q = enthalpy of water vapor
$r = q - 32$

q = rate of heat transfer
s = humid heat of air-water vapor mixture per pound of vapor free air
T = temperature of water, °F.
t = temperature of air, °F.
V = volume, cu. ft.
W = rate of water flow
λ = latent heat of vaporization of water

Subscripts

a = in air stream
1 = entering air, entering water
2 = leaving air, leaving water
s = at saturation
w = at wet bulb
d = at dew point

REFERENCES ON HUMIDIFICATION AND DEHUMIDIFICATION

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Qualitative insight into the factors determining k_g and k_L in the absorption process is supplied by recourse to the basic laws of diffusion. From these laws may be derived[1] for the steady state diffusion of one gas through a stagnant film of a second gas

$$m/A = \frac{b_g P}{RTBp_{BM}}(p_1 - p_2) = \frac{k}{Bp_{BM}}(p_1 - p_2) \tag{2}$$

where $p_1 - p_2$ is the partial pressure gradient of the diffusing gas across the stagnant film, b_g is the diffusion coefficient in volumetric units, and $k = b_g P/RT$.[2] The diffusion laws in liquids are less well known. With less certainty, by analogy with gaseous diffusion, there results for the steady state diffusion of a solute through a film of a stagnant solvent (1, 26)

$$m/A = \left[\frac{b_L}{B_L}\varphi(C_A, C_B)\right](C_2 - C_1) = \frac{b'_L}{B_L}(C_2 - C_1) \tag{3}$$

where $C_2 - C_1$ is the concentration gradient of the solute across the film and b'_L is a diffusion coefficient which depends upon the concentrations of solute, C_A, and solvent C_B, in the film.

In case diffusion through stagnant gas and liquid films were alone involved in the absorption process, it is seen that Equations (1) and (2) predict the gas film coefficient to be given directly by

$$k_g = \frac{b_g P}{RTBp_{BM}} = \frac{k}{Bp_{BM}} = \frac{k'_g}{p_{BM}} \tag{4}$$

Hence k_g should depend upon the diffusion coefficient of the solute gas, the mean partial pressure of the inert carrier gas in the film and the film thickness. The latter in turn depends upon the dynamic flow conditions in the apparatus and is some function of the Reynolds' number, $B = \varphi(Du\rho/\mu)$. Temperature affects k_g since the diffusion coefficient, viscosity, and density of the gas are dependent upon temperature. Similarly, by Equation (3), the diffusion coefficient in the liquid, the concentrations of solute and solvent in the film, and the turbulence conditions in the liquid phase should determine k_L.

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[1] See Sherwood, *Absorption and Extraction*, Chapter I.

[2] b_g is usually expressed as

$$\frac{(\text{cc})\,(\text{cm})}{(\text{sec})\,(\text{cm})^2}$$

in the scientific literature. The coefficient $k = b_g P/RT$ expresses the diffusion coefficient in molal units, such as

$$\frac{(\text{gm-mols})\,(\text{cm})}{(\text{sec})\,(\text{cm})^2}.$$

It may be expressed in other units as (gms) (cm)/(sec) (cm)^2, (lbs.) (ft.)/(hr) (sq. ft.) or (lb-mols) (ft.)/(hr.) (sq. ft.), by use of the proper conversion factors.

The theoretical diffusion relations apply rigorously only to diffusion through a completely stagnant film. In the actual absorption process the rate of transfer of the solute gas to the laminar film by convection or so-called "eddy diffusion" in the turbulent main fluid stream is involved, and the laminar film is in motion parallel to the interface. Insufficient is known concerning the mechanism of flow to permit a rigorous theoretical treatment of this complicated mechanism. Towle, Sherwood, and Seder (30) and Sherwood and Woertz (28) have, however, analyzed the role of eddy diffusion in mass transfer and report experimental studies of this subject.

For practical purposes it is customary to assume that the rate of transfer by eddy diffusion is also proportional to the partial pressure gradient and to write Equation (2) in terms of an "effective" or "equivalent" film thickness, B_x.

$$m/A = \frac{b_g P}{RTB_x p_{BM}} (p_g - p_i) \tag{5}$$

Experimental results are successfully treated in accordance with the above principles in this way.

Controlling Films. Inability to measure p_i and C_i in Equation (1) makes the direct experimental determination of k_g and k_L impossible except for k_g in the special case of the vaporization or condensation of pure liquids where the gas film alone is involved. Fortunately, conditions obtain in the absorption process which permits them to be determined indirectly in many cases of practical importance.

In the event that the dissolving gas is very soluble in the solvent, as ammonia in water, it may be shown (1, 32) that p_i may be essentially the same as the partial pressure of the gas in equilibrium with its concentration in the main body of the liquid, p_e. The resistance to absorption is almost solely in the gas film and the partial pressure gradient across this film is essentially the same as if no liquid film were present. Equation (1) may be written

$$m/A = k_g(p_g - p_e) \tag{6}$$

and the gas film is said to be controlling. Coefficients calculated on the basis of Equation (6) are practically identical with the true k_g.

In a corresponding manner, if the dissolving gas is very slightly soluble in the liquid, as oxygen or carbon dioxide in water, the liquid film tends to become controlling. Equation (1) may be written

$$m/A = k_L(C_e - C_L) \tag{7}$$

and coefficients thus calculated for slightly soluble gases should be practically identical with the true k_L.

The conclusion that one thus measures the true individual film coefficients is substantiated by the experimental evidence that conditions in the liquid film have negligible effect on the absorption of a very soluble gas and vice versa for slightly soluble gases. k_g and k_L as well as the solubility of the gas govern the relative resistance of the two films since

$$\frac{k_g}{k_L} = \frac{C_i - C_L}{p_g - p_i}$$

If k_g is very much greater than k_L, which may well be the case for very high turbulence conditions in the gas film, it is possible that both films may be of importance even though the gas is very soluble.

Where the gas is of intermediate solubility the resistance of both films must be considered and the film coefficients cannot be directly measured experimentally.

Factors Affecting k_g and k_L. As pointed out, the determination of absorption coefficients and their quantitative relation to the theoretical variables involved, at present rests on an experimental basis. The theoretical diffusion relations are, however, indispensable guides to the conduct of experiment and interpretation of data.

From examination of the predictions of the theoretical diffusion relations and the available data in wetted-wall towers, Sherwood (26) concludes that k_g is: (1) Proportional to the 0.56 power of the diffusion coefficient; (2) Proportional to approximately the 0.8 power of the gas mass velocity; (3) Proportional to approximately the 0.2 power of the tower diameter; (4) Independent of the total pressure at a given mass velocity; (5) Proportional to the square root of the absolute temperature at a given mass velocity; (6) Inversely proportional to the mean partial pressure of the inert gas, p_{BM}.

Employing the vaporization of liquids into air, in which case only the gas film is involved, Gilliland and Sherwood (17) have developed the following equation for k_g in the wetted-wall tower in the turbulent region

$$\frac{D}{B_x} = 0.023\left(\frac{Du\rho}{\mu}\right)^{0.83}\left(\frac{\mu}{\rho b_g}\right)^{0.44} \tag{8}$$

in which the group D/B_x is related to k_g by

$$\frac{D}{B_x} = \frac{k_g DRTp_{BM}}{b_g P} = \frac{(m/A)DRTp_{BM}}{\Delta p b_g P} \tag{9}$$

Δp is the overall partial pressure gradient, $p_g - p_e$, where p_g is the partial pressure of the solute gas in the main body and p_e its equilibrium partial pressure for the liquid phase. In the case of vaporization of pure liquid (no liquid film), p_e becomes p_i. Any self-consistent set of units may be employed in Equation (8). Its form is suggested by analogy with fluid friction and heat transfer (7, 10) and by dimensional analysis.

Gilliland (16, 32) points out that the method of correlating mass transfer data represented by equation (8) is theoretically incompatible with a concept of transfer in sequence through the turbulent core by eddy diffusion and the laminar film by simple diffusion; in the film the rate of transfer is proportional to the first power of the diffusivity, whereas transfer in the core by eddy diffusion should be independent of diffusivity.[3] He develops a method of calculating separately the resistances to mass transfer of the turbulent core and of the film, respectively, based on experimental measurements of k_g for each of two solute gases with different diffusivities. He also suggests a method of correlating gas film absorption coefficients which takes account of the different transfer mechanisms in the core and film.

Equation (8) may be expected to apply only when the gas film offers the only resistance to absorption and where the design of the tower is such that entrance turbulence is negligible. For short towers without calming sections, the equation must be corrected for entrance effects (13). The relation is useful for analyzing material transfer and absorption data obtained in laboratory experiments in properly designed wetted-wall towers. On a log-log plot of D/B_x versus $Du\rho/\mu$, data for any one gas-liquid system should give a straight line, and a similar plot of $(D/B_x)/(\mu/\rho b_g)^{0.44}$ versus $\left(\frac{Du\rho}{\mu}\right)$ should result in a single line for all systems. Sherwood (26), Gilliland and Sherwood (17), and Fallah (13) show such plots for the available data.

A method of treating data for k_g in wetted-wall towers in the viscous flow region is given by Gilliland and Sherwood (17) and further discussed by Sherwood (26).

In packed and spray towers no general correlation of the absorption coefficients with operating variables and physical properties of the system is yet possible.[4] Empirical equations and data for various types of such equipment and particular gas liquid systems may be found in the literature and in the various texts. These are summarized and discussed by Sherwood (26).

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[3] In this connection see references 28 and 30.

[4] In this connection, however, see the recent work of Sherwood and Holloway [papers presented before the 32nd Annual Meeting, A.I.Ch.E., Providence, Rhode Island, Nov. 17, 1939]. These authors report experimental data for the absorption of ammonia by air and sulfuric acid, the vaporization of water into air, and the desorption of carbon dioxide, oxygen, and hydrogen from water into air in packed towers with various conditions and packings and show that the liquid film absorption coefficients for the slightly soluble gases may be correlated by an expression analogous to equation (8). See also the work of Johnstone and Kleinschmidt [Trans. Am. Inst. Chem. Engrs., 34,181 (1938)] and of Johnstone and Williams [Ind. Eng. Chem., 31,993 (1939)]. These authors present theoretical and experimental studies of the absorption of gases in wet cyclone scrubbers and by falling liquid droplets. They develop theoretical relations for the rate of absorption and the absorption coefficient for liquid droplets in terms of physical properties, droplet size, and the dynamics and geometry of the apparatus and find approximate agreement of the theoretically derived relations with experiment.

The work of the above authors will be useful in connection with setting conditions and analyzing data for experimental laboratory studies of absorption in equipment of the types in question.

The information and data at hand are insufficient to warrant general conclusions concerning the factors which determine the liquid film coefficient, k_L.[4] Those available indicate that k_L depends upon the liquor velocity and relatively little upon the nature of the diffusing solute (19).

Overall Coefficient. To facilitate the practical use of Equation (1) two overall coefficients are defined and employed in terms of overall driving forces

$$\frac{m/A}{(p_g - p_e)} = K_g \tag{10}$$

and

$$\frac{m/A}{(C_e - C_L)} = K_L \tag{11}$$

It is evident that K_g and K_L become identical with the respective film coefficients when the gas or the liquid film controls.

Overall coefficients should not be used to calculate laboratory data unless Henry's Law is obeyed, or the equilibrium relation between partial pressure and concentration in the liquid is linear over the concentration range involved. Under other circumstances K_g and K_L calculated by Equations (10) and (11) apply only to the concentration conditions in a given experiment and must be regarded as essentially empirical.

When Henry's Law holds, K_g and K_L can be shown to be related to the film coefficients by the following equations:

$$\frac{1}{K_g} = \frac{1}{k_g} + \frac{H}{k_L} \tag{12}$$

$$\frac{1}{K_L} = \frac{1}{k_L} + \frac{1}{Hk_g} \tag{13}$$

and

$$HK_g = K_L$$

where H is the proportionality constant in the relation $p = HC$. $1/K_g$ and $1/K_L$ are regarded as the overall resistances and $1/K_g$, $1/Hk_g$, $1/k_L$, and H/k_L as the individual film resistances expressed in terms of pressures and concentrations, respectively. Unless k_g is inordinately large compared with k_L, for soluble gases H is small, the term H/k_L becomes negligible, and $1/K_g$ practically equals $1/k_g$.

Equations (12) and (13) are useful for the analysis of absorption data in terms of overall coefficients if the restrictions on the use of these pointed out above, are observed. Experiment shows that k_g is proportional to some power of the mass velocity of the gas, $k_g = \gamma g^n$, the value of the exponent, n, in most cases being approximately 0.8. Substituting for k_g we may write

$$\frac{1}{K_g} = \frac{1}{\gamma g^{0.8}} + \frac{H}{k_L} \tag{14}$$

and k_L may be assumed independent of gas velocity. If $1/K_g$ for a series of runs at different gas velocities be plotted versus $1/g^{0.8}$ as indicated by Equation (14) a straight line with positive intercept H/k_L and slope $1/\gamma$ should result. If the gas film entirely controls, H/k_L is negligible and the line passes approximately through the origin. A positive intercept indicates that the liquid film is involved. If the liquid film entirely controls, the line is horizontal since K_g is then independent of gas velocity. Experimental results agree with these conclusions.

The method is of considerable importance since it supplies essentially the only available experimental method of determining whether or not the gas or the liquid film entirely controls in a given absorption process and is successfully employed to test experimental data.

Cases in which k_g is proportional to a power of g other than the 0.8 may occur. In this event, values of n may be assumed until one is found which results in a straight line. Or, a log-log plot of K_g versus g, the slope of which is n, may first be made to determine the proper value of the exponent.

If conditions in the liquid film are maintained constant, in cases where both films control, it is possible to calculate k_g and k_L from the slope and intercept, respectively, of the plot $1/K_g$ versus $1/g^n$.

Equation (12) allows the calculation of K_g if values of k_g, k_L, and H are known. Although of potential importance, data on k_g and k_L are too few to give this method wide use. Instructive tests of absorption principles may, however, be made by comparing K_g observed for a given gas system in a wetted-wall tower with values calculated from experimental measurements of k_g for ammonia and k_L for carbon dioxide in the same apparatus. To this end, k_L is assumed to be the same for all solutes and k_g corrected to the particular set of conditions by use of the Sherwood and Gilliland equation (26)[5] with allowance for variations in p_{BM}. Reasonable agreement has been obtained from such calculations.

Capacity Coefficient. Equipment such as the packed or spray tower does not permit the direct measurement of the area of contact between liquid and gas. Absorption coefficients are calculated in such cases in terms of the effective volume V by introducing a new factor a, the specific contact area per unit of packed volume; where $a = A/V$. Applied to such a case, the absorption rate equation is written as

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[5] For this method see Sherwood, *Absorption and Extraction*, pp. 65-66 and 189-190.

$$m/V = K_g a(p_g - p_e) \tag{15}$$

or

$$m/V = K_L a(C_e - C_L) \tag{16}$$

$K_g a$ is the capacity coefficient expressed as mols of solute transferred per unit time per unit tower volume per unit pressure difference and $K_L a$ a similar coefficient expressed per unit concentration difference. English units are customarily employed for these coefficients.

In interpreting data for packed towers it should be borne in mind that a as well as K_g may be varied by changes in the operating variables. There is no known method of distinguishing the effect of these variables on K_g and on a. $K_g a$ and $K_L a$ depend upon the nature of the tower packing as well as upon the other variables previously discussed. The available data for capacity coefficients in packed towers are conveniently summarized in references (1), (23), (26), and (32); see also (6), (14), and (20).

The Transfer Unit and H.T.U. Chilton and Colburn (5) and Colburn (8) [see also references (9, 26), p. 346] have developed an alternate method of calculating transfer operations which may be conveniently employed for experimental absorption studies.

The absorption rate equation for transfer across the gas film only at any point in a continuously operated absorption tower is written

$$\frac{(p_{BM})(dp)}{(P - p_g)(p_g - p_i)} = \frac{k'_g a M_b (P - p_g) dZ}{P(G/S)} \tag{17}$$ [6]

and the left hand term which defines the difficulty of the transfer operation is defined in terms of a "number of gas film transfer units", N_g, or

$$dN_g = \frac{(p_{BM})(dp)}{(P - p_g)(p_g - p_i)} \tag{18a}$$

In dealing with this relation for the transfer unit various systems of units may be used as desired, but it is convenient to express concentrations in terms of mol fractions. In terms of mol fraction y of the solute gas equation (18a) becomes

$$dN_g = \frac{(1 - y)_f (dy)}{(1 - y)(y - y_i)} \tag{18b}$$

where y_i is the mol fraction of the solute gas at the interface, $y - y_i$, the mol fraction driving gradient over the gas film, and $(1 - y)_f$ is the log mean of $(1 - y)$ and $(1 - y_i)$. The gas film transfer unit $(\text{H.T.U.})_g$, or height equivalent to a gas film transfer unit, is defined to be

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[6] In equation (17) $k'_g a = k_g a(p_{BM})$.

$$dN_g = \frac{dZ}{(\text{H.T.U.})_g} = \frac{(p_{BM})(dp)}{(P - p_g)(p_g - p_i)} = \frac{(1 - y)_f(dy)}{(1 - y)(y - y_i)} \tag{19}$$

Similarly, a "number of liquid film transfer units" and a $(\text{H.T.U.})_L$ for transfer across the liquid film only are defined

$$dN_L = \frac{dZ}{(\text{H.T.U.})_L} = \frac{(1 - x)_f(dx)}{(1 - x)(x_i - x)} \tag{20}$$

where the x terms represent the corresponding quantities in terms of the mol fraction of solute gas in the liquid phase.

Since for practical purposes it is usually necessary to refer to the main body liquid concentration and thus replace y_i by y_e, there is defined an overall transfer unit, N_{0g}, and an overall $(\text{H.T.U.})_{0g}$ based on the change in gas concentration

$$dN_{0g} = \frac{dZ}{(\text{H.T.U.})_{0g}} = \frac{(1 - y)_f(dy)}{(1 - y)(y - y_e)} \tag{21a}$$

and similarly overall quantities, N_{0L}, and $(\text{H.T.U.})_{0L}$, based on change in liquid concentration

$$dN_{0L} = \frac{dZ}{(\text{H.T.U.})_{0L}} = \frac{(1 - x)_f(dx)}{(1 - x)(x_e - x)}\,. \tag{21b}$$

Where solute concentrations are not too large,[7] these overall H.T.U.'s can be shown to be related to the individual film H.T.U.'s by

$$(\text{H.T.U.})_{0g} = (\text{H.T.U.})_g + (\text{H.T.U.})_L(qG_M/L_M) \tag{22a}$$

$$(\text{H.T.U.})_{0L} = (\text{H.T.U.})_L + (\text{H.T.U.})_g(L_M/qG_M) \tag{22b}$$

and

$$(\text{H.T.U.})_{0g} = (\text{H.T.U.})_{0L}(qG_M/L_M) \tag{22c}$$

The term G_M/L_M is the molar gas-liquid ratio and q is the slope of the equilibrium curve, dy_e/dx, at any given point. The term (qG_M/L_M) is essentially equal to the ratio of the slope of the equilibrium curve to that of the operating line.

H.T.U. supplies a convenient measure of the performance of absorption towers, or conversely, in design, the required height of the tower is

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[7] The rigorous relation is

$$(\text{H.T.U.})_{0g} = (\text{H.T.U.})_g + (\text{H.T.U.})_L\,(qG_M/L_M)(1 - x)_f/(1 - y)_f$$

For low solute gas concentrations the ratio $(1 - x)_f/(1 - y)_f$ becomes essentially unity and may be neglected and the approximate expressions used with sufficient accuracy in the majority of experimental cases.

readily calculated in terms of transfer units and H.T.U., if values of the latter are available. Colburn suggests certain advantages of this method over the use of absorption rate coefficients. As compared with k_ga and k_La, $(\text{H.T.U.})_g$ and $(\text{H.T.U.})_L$ in general vary but little with widely varying rates of flow or even for different systems. Whereas individual values of q and G_M/L_M may vary many-fold, the term (qG_M/L_M) is, for many cases, practically constant. In addition, while the numerical values of the absorption coefficients depend on the numerous different systems of units employed, the value of H.T.U. has only one dimension, length, and the term (qG_M/L_M) is dimensionless. Transformations of the relation for H.T.U. into other systems of units is also relatively simple.

H.T.U. are related to the mass transfer absorption coefficients by the following:

$$(\text{H.T.U.})_g = \frac{G_M}{k_ga(1 - y)_f} \tag{23a}$$

$$(\text{H.T.U.})_L = \frac{L_M}{k_La(1 - x)_f} \tag{23b}$$

$$(\text{H.T.U.})_{0g} = \frac{G_M}{K_ga(1 - y)_f} \tag{23c}$$

$$(\text{H.T.U.})_{0L} = \frac{L_M}{K_La(1 - x)_f} \tag{23d}$$

Calculation of Absorption Coefficients

The calculation of coefficients from experimental data requires the use of terminal conditions for the apparatus and an integrated form of the rate or capacity equation. Rigorous integration for the general case is complicated but simpler methods have been developed permitting relatively accurate coefficients to be calculated when certain conditions apply. It is recommended that chemical engineering laboratory experiments in general be designed to comply with these conditions so that the following methods may be used.

I. *Henry's Law is not obeyed* but the gas and liquid phases are sufficiently dilute so that p is approximately proportional to the molal, or weight, ratio of solute to carrier gas in the gas phase and C to the molal, or weight, ratio of solute to solute-free solvent in the liquid phase. The volume change in the apparatus is inappreciable so that the mean partial pressure of carrier gas, p_{BM}, is approximately constant. The capacity coefficients, k_ga and k_La, remain constant through the tower.

If these conditions hold, Sherwood (26) has modified the graphical procedure of Walker, Lewis, and McAdams (32) to provide the following method.

G = weight of inert carrier gas per unit time.
L = weight of solute-free absorbing liquid per unit time.
X_t = weight of solute per unit weight of solute-free solvent in the absorbing liquid entering the tower.
X_b = weight of solute per unit weight of solute-free solvent in the absorbing liquid leaving the tower.
Y_t = weight of solute gas per unit weight of inert carrier gas in the gas stream leaving the tower.
Y_b = weight of solute gas per unit weight of inert carrier gas in the gas stream entering the tower.

A material balance around the tower is

$$L(X_b - X_t) = G(Y_b - Y_t) \tag{24}$$

Plotted on an X *vs* Y diagram this relation is a straight line called the operating line. The differential form of the capacity equation is written in the same units as

$$\begin{aligned} LdX = GdY &= M_a \alpha_{\text{av}} k_g a (Y - Y_e) dV \\ &= M_a \beta_{\text{av}} k_L a (X_e - X) dV \end{aligned} \tag{25}$$

α_{av} and β_{av} are proportionality factors which convert $(Y - Y_e)$ to $(p - p_e)$, and $(X_e - X)$ to $(C_e - C)$, respectively.

(*a*) *Gas film controls.* The integrated form of equation (25) which applies is

$$k_g a = \frac{G}{\alpha_{\text{av}} V M_a} \int_{Y_t}^{Y_b} \frac{dY}{(Y - Y_e)} \qquad (26)$$[8]

To calculate $k_g a$ from Equation (26) the right hand term is integrated graphically by plotting $1/Y - Y_e$ versus Y and taking the area under the curve between the limits Y_b and Y_t. Values of $Y - Y_e$ are calculated by plotting the equilibrium curve for the system on the X *vs* Y diagram and scaling off the vertical distances between it and the operating line for several values of X. α_{av} is taken as the arithmetic average for the two ends of the tower,

$$\alpha_{\text{av}} = \frac{1}{2}\left[\left(\frac{\Delta p}{\Delta Y}\right)_t + \left(\frac{\Delta p}{\Delta Y}\right)_b\right]$$

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[8] Differences in the exact form of this and the following relations are found in the literature. These arise from differences in the choice of units. The units here employed are those chosen by Sherwood and are probably of most general application inasmuch as the molecuar weight of the absorbing liquid or carrier gas may in cases be unknown.

(*b*) *Liquid film controls.* The integrated form of the rate equation (25) which applies is

$$k_La = \frac{L}{\beta_{av} V M_a} \int_{X_t}^{X_o} \frac{dX}{X_e - X} \tag{27}$$

k_La is calculated by graphical evaluation of the integral as before, in this case measuring the values of $X_e - X$ horizontally between the equilibrium curve and the operating line for various values of Y. β_{av} is the arithmetic average for the two ends of the tower,

$$\beta_{av} = \frac{1}{2}\left[\left(\frac{\Delta C}{\Delta X}\right)_t + \left(\frac{\Delta C}{\Delta X}\right)_b\right]$$

(*c*) *Neither film controls.* The theoretical methods, developed where the resistances of both films are involved, require an advance knowledge of k_g and k_L, and hence are of little value for the purpose of calculating coefficients from experimental data. If Henry's Law is not obeyed it is very difficult, if not impossible, to calculate theoretically significant coefficients. The analysis of data in such cases is largely empirical.

In this connection, correct use of Equations (4) or (26) implies that it may be legitimately assumed in advance or independently demonstrated that one or the other film controls for the case in question.

II. *Henry's Law holds*, or the equilibrium partial pressure of the gas is a linear function of its concentration in the liquid over the concentration range involved; p_{BM}, k_ga and k_La are essentially constant.

(*a*) *Gas film controls.* Overall coefficients may be employed and the integrated form of Equation (19) becomes

$$K_ga = \frac{G(Y_b - Y_t)}{M_a \alpha_{av} V (Y - Y_e)_{Lm}} \tag{28}$$

Or, the form

$$K_ga = \frac{m}{V(p - p_e)_{Lm}} \tag{29}$$

may be used. $(Y - Y_e)_{Lm}$ and $(p - p_e)_{Lm}$ are the logarithmic mean averages for the two ends of the tower

$$(Y - Y_e)_{Lm} = \frac{(\Delta Y)_b - (\Delta Y)_t}{2.3 \log \dfrac{(\Delta Y)_b}{(\Delta Y)_t}}$$

and

$$\Delta p_{Lm} = (p - p_e)_{Lm} = \frac{(\Delta p)_b - (\Delta p)_t}{2.3 \log \dfrac{(\Delta p)_b}{(\Delta p)_t}}$$

respectively. The equilibrium curve must be linear between X_t and X_b for the correct use of equations (28) and (29).

(*b*) *Liquid film controls.*—The integrated form of equation (27) becomes

$$K_La = \frac{L(X_b - X_t)}{M_a\beta_{\text{av}}V(X_e - X)_{\text{Lm}}} \tag{30}$$

or the form

$$K_La = \frac{m}{V(C_e - C)_{\text{Lm}}} \tag{31}$$

may be used. $(X_e - X)_{\text{Lm}}$ and $(C_e - C)_{\text{Lm}}$ are the logarithmic means of the driving force for the terminals of the tower in weight units and in concentration units, respectively. The correct use of Equations (30) and (31) requires that the equilibrium curve be linear between Y_t and Y_b.

(*c*) *Neither film controls.* In this case for the conditions postulated, Equations (28), (29), (30), and (31) may be used to calculate results in terms of overall coefficients.

III. *General Case.* Where the conditions specified above are not valid, Sherwood (26) presents a general method based upon a more rigorous integration of the rate equation. This requires only the assumption that the pressure drop due to friction in the apparatus be negligible compared with the total pressure. It allows for a variation of p_{BM} through the apparatus and may be modified to take account of possible variation in the film thickness, B_x, produced by varying gas velocity, if the relation between B_x and k_ga be known. Since this is seldom the case, this possibility is as yet of little assistance in analyzing laboratory data. The method is necessarily somewhat complex and laborious for routine application in the laboratory.

In this connection it is well to bear in mind the following facts. Reference to Equation (4) will show that the coefficient k_ga is given by

$$k_ga = \frac{k}{Bp_{BM}} = \frac{k_g'}{p_{BM}} \tag{32}$$

and hence is a function of the mean partial pressure of the inert carrier gas across the film and the film thickness. The latter varies with gas velocity. If the concentration of the solute gas is high and a sufficiently large fraction is absorbed, both p_{BM} and the gas velocity, and consequently k_ga, will vary through the tower. Values of k_ga calculated from data by the preceding equations may be approximately corrected for variation in p_{BM} by using an average value of this quantity to calculate a new coefficient k_g' as indicated by Equation (32). This coefficient may be expected to be constant provided the gas velocity does not vary too widely. It does not seem possible at present to allow for variation in the gas velocity through the tower but an average value calculated from the gas volumes at the terminals should be used.

E. M. Baker and J. H. Wiegand (3) have recently proposed a graphical method for absorption tower calculations which avoids the necessity for many of the assumptions made above and for an advance knowledge of the relative control of gas and liquid films. It may be applied to the calculation of absorption rate coefficients from experimental data in the general case but would appear to be unnecessarily rigorous and laborious for routine application under ordinary circumstances to undergraduate laboratory absorption data.

Alternate Methods of Measuring Performance

Plate Column. The performance of columns of the bubble plate type is usually measured in terms of a plate efficiency. This depends both upon the mechanical design of the plate and the operating variables. An average overall plate efficiency is calculated by dividing the number of theoretical plates required for the given absorption by the number of actual plates employed. Or, the definition of Murphree (22) may be used to calculate an individual plate efficiency. The required theoretical plates may be calculated by the well known graphical stepwise method (23, 26).

H.E.T.P. Particularly for practical application the performance of a packed tower may be measured by comparing it with a column of theoretical plates. This method defines an H.E.T.P. (24) as the *height of packing equivalent to one theoretical plate* and calculates the H.E.T.P. by dividing the packed height of the column by the number of theoretical plates required to effect the observed absorption.

H.E.T.P. measures column performance as do $K_g a$ and $K_L a$ and may be theoretically related (27) to these coefficients. It is less fundamental than these and depends upon the relative slopes of operating and equilibrium curves and hence may vary widely over the concentration range in a given column. It varies with the nature of the packing and with operating variables. Further, an accurate calculation of H.E.T.P. for a given packing requires that the column be equivalent to a relatively large number of theoretical plates. Correlation of H.E.T.P. with the pertinent variables has not been successfully accomplished and this method is primarily empirical.

H.T.U. Calculation of experimental absorption tower data in terms of H.T.U. requires a calculation of the number of transfer units by suitable integration of equation (18b), (20), (21a), or (21b) between the terminal conditions obtaining in any one experiment. The number of transfer units thus calculated is divided into the effective height of the tower used to obtain the corresponding H.T.U. value.

Except under special conditions, it will ordinarily be necessary in practice to calculate values in terms of overall H.T.U. by integration of equation (21a) or (21b). Overall H.T.U.'s are, in general, subject to restrictions similar to those imposed on the use and calculation of overall absorption rate coefficients, see page 311. If the term (qG_M/L_M) is not constant and the ratio $(1 - x)_f/(1 - y)_f$ is not essentially unity under a given set of experimental conditions, the overall H.T.U. is varying with concentration through the length of the tower and its calculated value has only empirical significance for this exact set of conditions. In order for the term (qG_M/L_M) to be constant, the slope of the equilibrium curve q must be constant; this is the case if Henry's Law is obeyed over the concentration range in a particular experiment, conditions are essentially isothermal, and G_M/L_M does not vary. For the ratio $(1 - x)_f/(1 - y)_f$ to approach unity, concentrations must not be too high in either phase. It is not usually difficult to devise experimental conditions so that these conditions are maintained.

In the event that experimental conditions are such that it is known or can be ascertained in advance that the gas film alone is involved or entirely controls, equations (18b) and (21a) become identical and the true gas film values of N_g and $(\text{H.T.U.})_g$ are calculated by proper integration of either. In this case $(\text{H.T.U.})_g$ will be in a given experiment constant through the tower independent of concentration and has the related theoretical significance. A similar situation exists when the liquid film only is involved or entirely controls.

Methods of integration for the calculation of N_g and N_{0g} are illustrated below. They are more fully considered in references (5), (8), and (9). Similar procedures may be employed for N_L and N_{0L}.

Under special conditions analytical integration is feasible.

1. If Henry's Law is obeyed, the term (qG_M/L_M) is constant, and concentrations are sufficiently low so that the ratio $(1 - x)_f/(1 - y)_f$ is essentially unity, then equation (21a) integrates to

$$N_{0g} = \frac{y_b - y_t}{(y - y_e)_{Lm}} \tag{33}$$

where $(y - y_e)_{Lm}$ = log mean of $(y - y_e)_t$ and $(y - y_e)_b$.

2. If Henry's Law is obeyed and the liquor fed contains no solute, integration of equation (21a), assuming the ratio $(l - x)_f/(l - y)_f$ to be unity, gives

$$N_{0g} = \frac{2.3}{(1 - qG_M/L_M)} \log \left[(1 - qG_M/L_M)(y_b/y_t) + qG_M/L_M \right] \tag{34}$$

3. In a third case, if there is no back pressure of solute gas from the

liquid, y_e is zero, the gas film controls entirely, and equation (18b) intergrates rigorously to

$$N_g = \int_t^b \frac{(1-y)_f(dy)}{(1-y)(y)} = \log_e \left[\frac{\log_e (1-y_t)}{\log_e (1-y_b)}\right] \tag{35}$$

If further, absorption is nearly complete so that y_t is small relative to unity, then equation (35) reduces approximately to

$$N_g = \log_e \left(\frac{y_b}{y_t}\right) \tag{35a}$$

4. In the general case where the above simplifications are not justified and Henry's Law is not followed, graphical integration is necessary. Equation (18b) may be integrated graphically in a manner analogous to that employed for equation (26) for the absorption coefficient when there is only a gas film to be considered; or equation (20) is integrated as in the case for equation (27) when only a liquid film need be considered. If both films are involved and Henry's Law is not valid, the case is the same as that for calculating absorption rate coefficients as pointed out on page 313. In order to use the theoretical integration methods advance knowledge of $(\text{H.T.U.})_g$ and $(\text{H.T.U.})_L$ is necessary.

Colburn (8) has reviewed the available absorption data in terms of H.T.U.'s. He also suggests methods of determining the relative importance of gas and liquid film from H.T.U. measurements, of estimating film H.T.U. values from overall H.T.U.'s, and of correlating H.T.U. with properties and operating variables.

It may be seen from equation (22a) that if conditions are such that the term (qG_M/L_M) is very small, then $(\text{H.T.U.})_{og}$ becomes equal approximately to $(\text{H.T.U.})_g$ and hence the desirability of carrying out experimental studies under such circumstances.

In contrast to H.E.T.P. the H.T.U. has the fundamental basis of the absorption rate coefficient.

Baker (3) has proposed a convenient graphical stepwise method for calculating H.T.U.

Pressure Drop and Flooding Velocity

The pressure drop due to friction in packed towers depends upon the type and dimensions of the packing, the physical properties and velocity of the gas, and the rate of liquor circulation. At sufficiently high gas velocities at a given liquor circulation, the pressure drop rises almost vertically with velocity, the liquor begins to be blown back out of the top of the tower with the gas and the column is said to "flood." This flooding point depends upon both gas and liquor flows.

For methods of treating frictional pressure drop and flooding in packed towers and data on these factors, reference should be made to the chapter on Fluid Flow, bibliography references (1), (23), and (26), and to the literature.

Chilton and Colburn (4), Mach (21), Uchida and Fujita (31), and White (33) have studied pressure drop in towers of various diameters and types and with several sizes of packing. Equations and correlations of data for pressure drop in dry packings and with liquor circulation are presented by these authors. In general these involve a log-log plot of a modified friction factor, f^*, versus a modified Reynolds number, $(d_p u_o \rho)/\mu$, where d_p is the packing diameter and u_o the superficial gas velocity based on the total tower cross section. Data on flooding velocities and liquid holdup, methods of treating and plotting such data, and correlations of flooding velocity measurements are reported by Mach, Uchida and Fujita, White, Colburn (9), Robinson and Gilliland (25), Elgin and Weiss (12), and by Sherwood, Shipley, and Holloway (29). The latter authors propose a correlation method for flooding velocity which utilizes dimensionless functions of gas and liquor velocities and packing dimensions and show a curve for such a correlation. Elgin and Weiss have coordinated pressure drop, liquid holdup, and flooding velocity from both visual observations and measurements. Colburn has reviewed the available data for pressure drop and flooding velocity and suggests a method of plotting the latter. It is suggested that the results of laboratory studies of these quantities be treated, plotted, and compared with the relations and data of the above workers.

Laboratory Study of Gas Absorption

Four principal types of absorption towers can be employed for laboratory experiments. It is recommended, however, that either the wetted-wall or packed towers be used.

Wetted-wall Tower. The gas flow may be either parallel or countercurrent to the descending stream of liquid on the walls. Countercurrent flow is the normal case. Flow conditions in the gas phase may be defined in terms of the Reynolds number. In the liquid film descending on the walls, conditions are less certain, particularly at high rates of flow. At such rates of liquor flow and with columns of large diameter, wave motion occurs in the descending film. The mechanism of this phenomenon is as yet unexplained. Except for a slight alteration of the contact area it should not be of importance if the resistance of the liquid film is negligible. Whether or not the liquid film is in turbulent or viscous flow may be approximately estimated from the data and plot of Cooper, Drew, and McAdams (11), who conclude that flow in thin liquid layers may be safely considered as viscous below $Re = 2100$.

Since it is imperative that the tower wall be completely wetted by the liquid at all times, such towers are most satisfactorily constructed of glass, permitting visual control of this factor. The diameter of tower employed must be governed by available facilities and should be such, relative to the volume of gas flow available, that operation well into the turbulent region is permitted. A common mistake with such equipment is the use of diameters and gas velocities such that the flow is well in the viscous region and the attempting of theoretical analysis of results without ascertaining the flow condition involved. Diameters of 2 to 3 inches are probably most satisfactory for ordinary laboratory conditions but smaller towers may be dictated by necessity.

The height of the tower employed, determined by the gas-liquid system under study, entering concentration of solute gas, and the desired flow range must be such that measurable absorption is obtained but equilibrium never attained. It is also well to adjust the height to the other conditions to be employed so that the pressure drop and the fraction of solute gas absorbed are not too large.

The effect of entrance on the turbulent conditions in such towers should be minimized by the provision of a calming section and design of the entrance.

The range of gas and liquor flows permitted by a given tower diameter is definitely limited, the two rates of flow being interdependent in this respect. At sufficiently high gas velocities the tower may flood and a portion of the liquid be carried out the top by the gas stream, or the flow of liquid down the tower may be entirely prevented. The permissible flow ranges must be determined experimentally for individual towers.

Packed Towers. These are useful for general gas absorption studies. Materials and details of construction may vary widely to fit the individual case.

"Wall effect" factors become appreciable in small diameter towers, and the tower diameter employed should be sufficiently large, consistent with the volume of gas and liquid flow available, to reduce them to a minimum. Preferably the diameter should not be less than 4 to 8 inches, and 18 to 24 inches is probably needlessly large for the average case. The ratio of the tower diameter to that of the packing employed should be at least 6 to 1 and preferably greater.

In designing the tower it is well to estimate in advance the volume of gas and liquor which is likely to be available, selecting a diameter which allows operation well into the turbulent range for the gas and insures proper wetting and distribution of the liquor in the packing. Diameters which provide for operation up to the loading and flooding velocities for the packing increase the utility of a tower.

In dealing with gas flow in towers with the packing dry, a modified Reynolds Number $d_p u_o \rho/\mu$, where d_p is the nominal average packing diameter, and u_o is the linear gas velocity based on the empty tower, has been successfully employed for friction correlations for solid packings, Raschig rings and Berl saddles. The region in which the flow will fall may be approximately estimated from the work of Chilton and Colburn (4), who find for this case: Re' (critical) $= d_p u_o \rho / \mu = 40\text{-}80$. White (33) reports for Raschig rings and for Berl saddles a value of Re' (critical) falling roughly between 25 and 50. Under room conditions for a 6 inch diameter tower packed with 1 inch diameter Raschig rings or Berl saddles, a gas flow of 65 cubic feet per *hour* corresponds to a linear gas velocity of 0.1 foot per second on the empty tower cross section and to a value of Re' of approximately 50 for air and a dry packing.

The height of the tower used will depend upon the gas-liquid system, entering solute gas concentration and the flow rates as pointed out previously. Arrangement of the tower to permit its height to be varied readily offers obvious advantages. Inclusion of provision for pressure drop measurement multiplies its uses. Provision for proper distribution of the liquor at its point of entrance should be made. A tower constructed from Pyrex glass pipe sections has been found to afford a number of instructional advantages.

Packings of a wide variety of types and construction materials are possible. The authors favor use of simpler forms of the more or less standardized modern types, such as commercial clay, porcelain, or carbon Raschig rings or Berl saddles. The characteristics of such packings and data on their properties are, in general, known. Since ½ inch diameters are usually the smallest commercially obtainable, a minimum column diameter of 3 inches is indicated.

It should be borne in mind that properties of commercial packings such as weight, surface area, and free space per unit tower volume may vary considerably when used in small experimental towers from manufacturers' specifications for these quantities. Hence, if quantitative treatment and interpretation of data obtained with them are to be attempted it is well to measure such properties on the packing as actually used in the tower.

Plate and Spray Towers. Towers of these types are less useful for laboratory experiments than those above, but experiments designed to study their performance may be readily carried out. For this purpose, the authors have found that useful small plate columns having 3 to 6 plates may be readily and inexpensively constructed of 6 to 8 inch Pyrex pipe sections and small diameter, commercially obtainable, bubble caps.

The proper design and operation of spray type towers in the laboratory are involved, and the results must be interpreted primarily on an empirical basis. For information concerning this type of equipment see, however, the recent work of Hixson and Scott (18), and of Johnstone and Kleinschmidt, and Johnstone and Williams, footnote, p. 307.

Systems for Study

The selection of a suitable carrier gas-solute gas-liquid system for studies in the undergraduate laboratory offers a number of difficulties, the range of choice being considerably narrowed by certain requirements. The authors know of no system completely satisfactory from every point of view.

The vaporization or condensation of pure liquids into air or other inert gases affords a readily used and relatively simple means for investigating the principles of material transfer, but involves only the gas film.

An ideal absorption system from the laboratory viewpoint would be one for which: solute and carrier gas mixtures are available at low cost and in steady volumes of flow sufficient to permit use of a range of gas concentrations and velocities, equipment of suitable size, and runs of length sufficient to insure attainment of a "steady state"; an inexpensive absorbing liquid, introducing no storage or recovery problems, is required in not excessive quantity; no corrosion problems enter; easy routine analysis of both gas and liquid phases is possible; heat of solution is low; the gas mixture is not inflammable or explosive; Henry's Law approximately applies, or one or the other film is entirely controlling; and equilibrium data at a series of temperatures are available or readily determined.

Air is by far the most satisfactory carrier gas. The solute gas, except perhaps for the existence of special facilities or conditions, is limited to inexpensive, readily obtainable, tank gases, or to vapors of not too high boiling liquids added to the carrier gas stream. Suitable tank gases are practically confined to ammonia, sulfur dioxide, and carbon dioxide. With these, for appreciable flows, arrangements for supplying the heat of vaporization of the liquefied gas must be provided to insure steady flow. The cost of gas for large scale operation becomes also an appreciable item. Various vapors, as benzene, acetone, carbon tetrachloride, or water, are possible, but on account of fire and explosion hazards, the authors prefer the last two if the laboratory operation is on a large scale.

Water provides the most satisfactory absorbing liquid. For the absorption of benzene or carbon tetrachloride an hydrocarbon oil is necessary. Analytical problems are immediately introduced and routine analysis of the liquid phase may become impractical. With carbon tetrachloride, analysis of the gas phase by means of a gas density balance, as practiced at the Drexel Institute (see below) is recommended. Storage problems and the cost of oil will generally introduce the necessity for simultaneously stripping and recycling the solvent. For the absorption of water vapor, solutions of calcium chloride, sulfuric acid, or others of low vapor pressure, can be used. The first is preferred from the viewpoint of corrosion. Absorption in an absorbing liquid involving chemical reaction with a dissolved solute, e.g., carbon dioxide by sodium hydroxide or carbonate, should in general be avoided. The complications introduced by secondary chemical reaction, the effect of the concentration of the solute, and difficulties in evaluating the true interfacial-equilibrium partial pressure of the gas introduce uncertainties in calculation and interpretation which overbalance any advantages gained from their use.

PART II. EXPERIMENTAL PROGRAM

As previously pointed out, gas absorption theory is based upon diffusion of the solute through gas and liquid films. In order to investigate the principles of such diffusional processes, a wetted-wall tower will be used in the first group of experiments, which are termed "Experiments in the Principles of Material Transfer." The first three of these experiments are concerned with vaporization of pure liquids. In this classification, the problem has been simplified by the elimination of one film, guaranteeing that the diffusional process is entirely limited to passage of the material through the other film. This is readily accomplished by the use of a pure liquid phase, all diffusion being confined to the diffusion of the vapor of this pure liquid into the gas stream. The remaining two experiments in this section study the absorption process. The first deals with the absorption of a soluble gas, in which case it has already been stated that the gas film is the controlling resistance to absorption, or that the main resistance of the diffusional process occurs in this phase. The opposite case, that of a very slightly soluble gas, has been omitted. It is particularly difficult, under such conditions, to measure accurately the variation in solute content of the gas and liquid phases from inlet to outlet. The inaccuracies thus introduced make it practically impossible to obtain data of any value in undergraduate work. The third case, and the most complicated theoretically, that of a moderately soluble gas, *is* adaptable to ordinary laboratory technique and consequently has been included to complete this section. Experiments, in similar equipment, may be readily designed to study the effect of inert gas (e.g., H_2, N_2). These are less adaptable to a student laboratory.

A complete experimental program on gas absorption requires the inclusion of equipment of more industrial significance than the wetted-wall columns. To cover such cases a second general section has been introduced. Intelligent operation of packed towers requires a knowledge of the operating characteristics of such equipment. Of fundamental importance are pressure drop and flooding, including their dependence on the rate of flow of gas and liquid. The first experiment in this section is designed to cover these phases of the problem. This is followed by two experiments dealing with absorption in equipment of the same general type. Experiment No. 50 is selected as offering an excellent illustration of the performance and operating characteristics of an absorption-stripping unit of more nearly commercial size. The authors are indebted to the Department of Chemical Engineering at Drexel Institute for permission to include this experiment. It is believed that certain changes suggested in Experiment No. 50 would increase its value and utility for general laboratory use.

Experiments are readily designed for study of the operation of spray and bubble cap columns. In the opinion of the authors, these are less desirable than the type presented. Composition of such experiments may follow the general method employed in "Gas Absorption in Packed Towers," particular emphasis being laid upon material balances and individual, or overall, plate efficiencies in the case of a bubble cap column (24).

It is to be understood that the material of the experiments detailed cannot be completely exhaustive. Alternate methods, calculations, and comparisons of the results with the work of unlisted investigators may well be employed. Such variations are left to the discretion of individual instructors. The students will be expected to draw *general* conclusions from their experimental work, as a regular portion of any report. These should include such items as discussion of the failure of results to check with previous experimenters, inadequacy of the equipment in design or operation, and sources of error noted during operation.

In practically all cases, units are unspecified. Any consistent system may be employed providing no change is made in the course of an experiment. The use of English units is, of course, customary. It is important to note that absorption coefficients are, in this chapter, reported entirely in mols, *not weight,* absorbed.

In all cases, all data taken in any one run are to be averaged. Calculations are to be made from these average results, for the period of operation.

NOMENCLATURE

a = area of contact per unit of packed volume
A = area of contact
b_g = diffusion coefficient of the gas in volumetric units (volume × length)/(time × area)
b_L = diffusion coefficient of the solute through the liquid film, in volumetric units
B = true thickness of a fluid film
B_x = "effective thickness" of the gas film

C, C_L = concentration of the solute in the main body of the liquid, mols/unit volume
C_e = concentration of the solute in the liquid in equilibrium with p_g, mols/unit volume
C.F.M. = cubic feet per minute
d_p = nominal packing size
D = tower diameter
f = friction factor
f^* = modified friction factor
F_A = White's correction factor for Fanning equation, packed towers
g = mass velocity = $u\rho$
G = weight of inert carrier gas per unit time
G_M = molar gas velocity, lb. mols/(hr.)(sq. ft.)
H = Henry's law constant = p/C
H.E.T.P. = height equivalent to one theoretical plate
H.T.U. = height of a transfer unit, ft.
k = diffusion coefficient, $b_g P/RT$, i.e. molal diffusivity
k_g = gas film absorption coefficient, mols/(unit area) (unit time) (unit partial pressure difference)
k_g' = a film coefficient = k/B_x
k_h = Henry's law constant = Y/X
k_L = liquid film absorption coefficient, mols/(unit area) (unit time) (unit concentration difference)
$k_g a$ = gas film absorption coefficient, mols/(unit volume) (unit time) (unit partial pressure difference)
$k_L a$ = liquid film absorption coefficient, mols/(unit volume) (unit time) (unit concentration difference)
K_g = overall absorption coefficient, mols/(unit area) (unit time) (unit partial pressure difference)
K_L = overall absorption coefficient, mols/(unit area) (unit time) (unit concentration difference)
$K_g a$ = overall absorption coefficient, mols/(unit volume) (unit time) (unit partial pressure difference)
$K_L a$ = overall absorption coefficient, mols/(unit volume) (unit time) (unit concentration difference)
L = weight of solute-free absorbing fluid per unit time
L_M = molar liquid velocity, lb. mols/(hr.)(sq. ft.)
m = mols of solute absorbed per unit time
M_a = molecular weight of solute gas
M_b = average molecular weight of inert (carrier) gas
N = number of transfer units, Z/H.T.U.
p_e = partial pressure of the solute gas in the gas phase in equilibrium with the concentration of solute in the main body of the liquid phase

p, p_g = partial pressure of the solute gas in the main body of the gas phase
p_{BM} = *log mean* partial pressure of the inert gas *in the gas film*
P = total pressure
q = slope of equilibrium x *vs.* y curve, dy_e/dx
R = gas constant in simple gas law.
Re = Reynolds number
Re' = White's modification of Re for packed towers = $d_p u_o \rho/\mu$
S = cross-sectional area of the tower
T = absolute temperature
u = linear gas velocity, actual
u_o = linear gas velocity, based on empty cross section, packed towers
V = packed volume
x = mol fraction of solute in the liquid; $(1 - x)$ is the mol fraction of solute-free solvent liquid
x_e = equilibrium value of x corresponding to y
X = weight ratio of solute to solute-free absorbing liquor
X' = mol ratio of solute to solute-free absorbing liquor
X_e = weight ratio of solute to solute-free absorbing liquor in equilibrium with the concentration of solute in the main gas body
y = mol fraction of solute in the gas; $(1 - y)$ is the mol fraction of inert gas
y_e = equilibrium value of y corresponding to x
Y = weight ratio of solute gas to inert gas
Y' = mol ratio of solute gas to inert gas
Y_e = weight ratio of solute gas to inert gas, in equilibrium with a concentration of solute in the main body of the liquid phase
$\Delta C = C_e - C_L$ = overall concentration gradient
$\Delta p = p_g - p_e$ = overall partial pressure gradient.
Δp_{Lm} = log mean of overall partial pressure gradient, over entire apparatus
Z = height of tower, ft.
$(1 - x)_f$ = log mean of $(1 - x)$ and $(1 - x_e)$
$(1 - y)_f$ = log mean of $(1 - y)$ and $(1 - y_e)$
α = proportionality factor = $\Delta p/\Delta Y$
β = proportionality factor = $\Delta C/\Delta X$
γ = proportionality factor = k_g/g^n
μ = absolute viscosity of gas phase, average
ρ = density of gas phase, average.

GENERAL SUBSCRIPTS

1, 2 referring to two parallel planes in the film, taken at right angles to the direction of diffusion
A, B referring to the solute and the solvent (or inert gas), respectively
b, t referring to conditions at the bottom and the top of the column, respectively
g—gas or gas film
i—at the interface
L—liquid or liquid film

PRINCIPLES OF VAPORIZATION IN WETTED WALL TOWERS

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EXPERIMENT NO. 43

Vaporization of Water

Object. 1. To determine, experimentally, coefficients of material transfer.

2. To study the effect of variation of rate of gas flow upon these coefficients.

3. To correlate experimental results by methods suggested in the literature.

Equipment. Figures 1 and 2 represent a wetted-wall tower which has, in the authors' laboratory, proven its adaptability to experiments of this nature. The transfer section is of glass, 2 inches in diameter, constructed of a 4 foot section. Liquid is introduced at the top of the

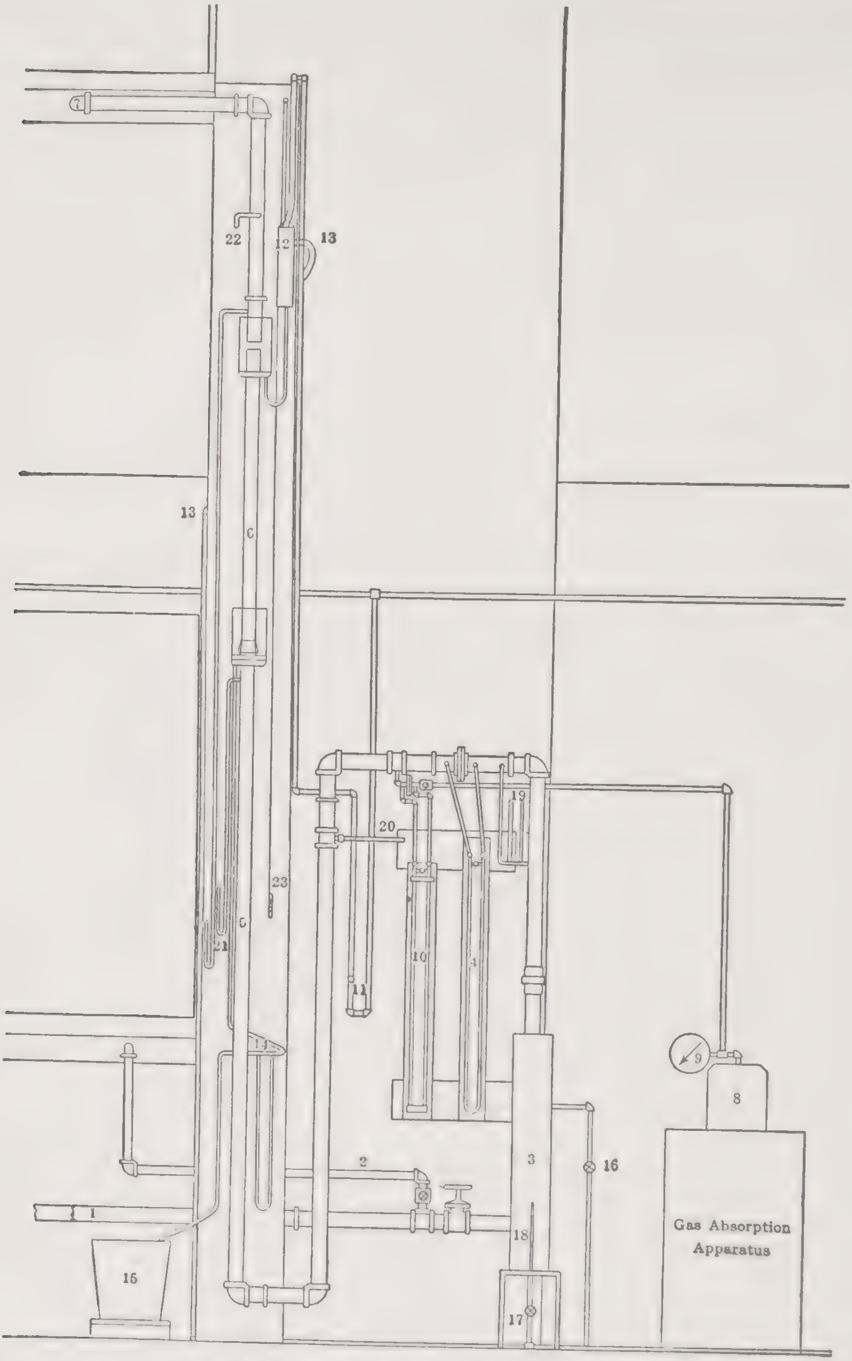

Fig. 1.—Gas Absorption Apparatus.

section which acts as a circular weir and distributes the film evenly over the clean walls. A slightly belled lower end removes the liquid at the bottom of the glass section, discharging into a chamber of annular cross section. A liquor seal is provided in this section by the height at which level of liquid is maintained. A calming section, of approximately 50 diameters of standard 2 inch iron pipe, serves to eliminate abnormal turbulence before the gas enters the glass section. As noted in Fig. 1, items 4, 8, 9, and 22 are not essential to the operation of the equipment for vaporization of pure liquids. Items 3, 16, and 17 are also unnecessary for the experiments listed in this section.

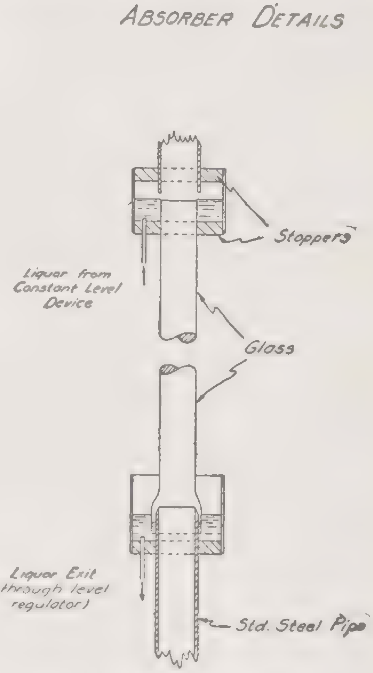
ABSORBER DETAILS
Stoppers
Liquor from Constant Level Device
Glass
Liquor Exit (through level regulator)
Std. Steel Pipe

Fig. 2.—Absorber Details.

Procedure. The gas phase shall be air and the liquid phase water. For this system, wet and dry bulb thermometer sets are the simplest analytical device. Water should be provided at approximately the same temperature as the air, the latter to be taken as it is supplied by the blower.

Four runs shall be made, the rate of gas flow being the variable. The air flows shall correspond to Reynolds Numbers (*Re*) of 7,000, 10,000, 14,000, and 20,000. The water rate shall be approximately 1 cubic foot per hour.

Prior to the laboratory period: Obtain the necessary calibrations for the air and water meters. Calculate the instrument readings required to give the Reynolds Numbers, specified above, for the air flow, and to give the specified water rate.

In the laboratory: **Part a:** Regulate the water flow to the tower so that the water meter shows the previously calculated reading. Start the blower with the air by-passing the column. Increase the air flow to the wetted-wall section by regulation of the inlet valve until one of the previously calculated air rates is shown by the meter. Allow the column to operate for at least 15 minutes to assure the production of equilibrium conditions. When the readings of manometers, thermometers, and humidity instruments indicate the attainment of a steady state, take readings of the data listed below. The run shall be of 20 minutes duration.

Repeat the experiment, varying the air velocity in subsequent runs to include the remaining three specified Reynolds Numbers.

Part b: Choosing one of the air rates previously investigated, make two more runs in which the air flow is maintained constant and water is charged to the tower at rates of 1.5 and 2.0 cubic feet per hour respectively. The data required for these additional runs are the same as those listed below for the runs of Part a.

Data

1. Temperatures.
    a. Water entering transfer section.
    b. Water leaving transfer section.
    c. Air entering transfer section, wet and dry bulb.
    d. Air leaving transfer section, wet and dry bulb.
2. Air orifice reading.
3. Water orifice reading.
4. Time to collect a given weight of water discharged from the tower (as a check on the flow meter).
5. Absolute pressures at top and bottom of the transfer section.
6. Barometric pressure.
7. Exact dimensions of material transfer section.

REPORT

A. *Calculate for each run, tabulating results:*
1. Apparent actual Reynolds Numbers of air flows used.
2. Actual water rate, weight per (unit time × unit cross section of the tower).
3. Mols of water vaporized per unit time.
4. Interfacial area.
5. k_g
6. D/B_x[9]
7. $\mu/\rho b_g$. (15, 17).

B. *Plot:*
1. D/B_x vs $Du\rho/\mu$, log-log.
2. k_g vs g, log-log.

C. *Discussion:*
1. From plot 1, determine graphically the form of the equation expressing the variation of D/B_x, calculated from the experimental data, with respect to $Du\rho/\mu$. Why is this simplified plot used to replace the general form, $(D/B_x)/(\mu/\rho b_g)^{0.44}$ vs $Du\rho/\mu$, recommended for all work on the vaporization of pure liquids?

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[9] In using Equation (9) for this calculation, the value of b_g must be obtained from the literature (15, 17). The term Δp becomes Δp_{Lm}, since variable conditions exist from one end of the absorber to the other. p_{BM} is calculated by the expression $p_{BM} = p_{B1} - p_{B2}/\ln\,(p_{B1}/p_{B2})$, where p_{B1} and p_{B2} are the average partial pressures of the inert gas film at gas inlet and outlet, respectively. If p_{B2} is nearly equal to p_{B1}, an arithmetic average, p_{Bav}, may replace p_{BM} as illustrated in Sherwood (26), page 41.

2. From plot 2, determine graphically the form of the equation expressing k_g in terms of g.
3. Discuss the significance of the equations determined in parts 1 and 2 of this discussion, comparing the constants obtained with Equation (8) of this chapter.
4. Explain, qualitatively, the observed effect of water rate, in terms of the accepted theory.
5. From the data on the air-water system, predict the value of k_g for an air-toluene system, the air rate corresponding to a Reynolds Number of 13,000 in each case.

EXPERIMENT NO. 44

Vapors Other Than Water as Diffusing Component

Object. 1. To study the effect on the rate of material transfer of variation of the physical properties of the vapor.

2. To compare experimental results with those in the literature.

Equipment. Same as Experiment No. 43.

Since liquids other than water are to be used in this experiment, certain minor changes are necessary in the equipment shown in Figure 1. To avoid the requirement of excessive quantities of these materials, a liquid reservoir should be provided. This should be accompanied by the installation of a pump for circulating the liquor. Wet and dry bulb thermometry as a means of analyzing the gas stream is also impractical under the conditions specified. The device suggested for the measurement of the rate of vaporization is one whereby the drop in level in the liquid reservoir may be measured. Constancy of rate of liquor input to the tower is essential for the success of such a method.

Procedure. The liquids suggested for this experiment are water, isopropyl alcohol, toluene, and secondary amyl alcohol. The rate of feed to the tower should be one cubic foot per hour in all cases. The rate of vaporization of each liquid is to be investigated at three air flows corresponding to Reynolds Numbers of 7,000, 13,000, and 20,000.

Prior to the laboratory period: See Experiment No. 43.

In the laboratory: See Experiment No. 43.

When changing liquids exercise special care toward the complete elimination from the system of the liquid on which experiments have just been completed.

DATA

See Experiment No. 43. Omit: Wet bulbs in part 1*c* and 1*d*. Add:

8. Level of liquor in reservoir tank.

REPORT

A. *Calculations:* See Experiment No. 43.

B. *Plot:*

1. D/B_x vs $Du\rho/\mu$, log-log, noting, by variation of the type of mark chosen as indications of experimental points, which refer to each liquid employed. Draw an average line through the points, for each liquid.
2. D/B_x vs $\mu/\rho b_g$ at constant values of $Du\rho/\mu$.

C. *Discussion:*

1. From plots 1 and 2, determine the constants which you obtain, experimentally, in the equation expressing the variation of D/B_x, in terms of $\mu/\rho b_g$ and $Du\rho/\mu$. Compare this equation with (8) of this chapter.
2. Compare the value of k_g for toluene as the wall liquid, predicted by calculation in Experiment No. 43, with that obtained experimentally.

EXPERIMENT NO. 45

GASES OTHER THAN AIR

Object. 1. To study the effect on the rate of material transfer of variation of the physical properties of the inert gas.

2. To compare experimental results with those in the literature.

Equipment. See Experiment No. 44.

Procedure. Air, hydrogen, and carbon dioxide shall be used successively as inert gases. In each case the flows of these materials shall be so adjusted as to produce Reynolds Numbers of 5,000, 8,000, and 12,000 in the transfer section. The wall liquid is to be water, being charged to the transfer section at a rate of one cubic foot per hour.

DATA AND REPORT: See Experiment No. 44.

PRINCIPLES OF GAS ABSORPTION

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EXPERIMENT NO. 46

VERY SOLUBLE SOLUTE GAS

Object. 1. To study the effect of variation of gas flow rate on the coefficients of absorption, using a very soluble gas.

2. To calculate the H.T.U. of the system at each rate of gas flow.

3. To compare the results with the work of previous experimenters.

Equipment. Employ a wetted wall tower of design similar to that shown in Figures 1 and 2. The entire equipment is used as shown.

Procedure. Titrations on the liquor leaving the absorber shall be used as a measure of the quantity of gas absorbed. In order to check the data so obtained it is advisable to make analyses of the gas entering and leaving the absorber. Due to low concentrations of solute gas and/or small percentages of the solute gas absorbed at high flow rates, ordinary gas analysis equipment are too inaccurate for this task. The incorporation of an absorption train, in series with and preceding, a wet gas meter is recommended. An excess of absorbing solution may be placed in the bottles of the train. After a measured quantity of inert has been drawn through the meter, the excess is titrated, and the calculated quantity of solute gas used to determine the concentration in the exit gas stream. By this method, material balance checks of 10% or better may be obtained.

Water is to be the absorbing liquid. Ammonia shall be the solute gas, air the inert. This mixture shall contain 5 per cent ammonia. The gas velocities shall be such that the Reynolds Numbers in the various runs shall be 7,000, 10,000, 14,000, and 20,000, respectively. The water rate shall be one cubic foot per minute.

Prior to the laboratory period: Obtain the calibration of the air, ammonia, and water meters. Calculate the instrument readings required to give the Reynolds Numbers, specified above, for gas flows, and to give the specified water rate.

In the laboratory: Adjust the water rate to give the required meter reading. Start the blower and gradually bring the air flow up to the required value. Feed water to the humidifier spray at such a rate that the air may be saturated at the temperature at which it enters the absorber. This rate of water flow is dictated by the wet-dry bulb thermometer readings as the air leaves the humidifier and by the temperature of the main gas stream measured after the point of addition of the ammonia. When the tower is operating satisfactorily under these conditions, admit ammonia to the air stream, adjusting the flow to the specified meter reading by a gradual increase in the valve opening.

Allow the apparatus to operate for at least 15 minutes before taking any data. Then, at 5 minute intervals, make readings of all the data listed below, continuing the run until these data show that equilibrium conditions have been maintained for a 15-minute period. In making calculations use only the data obtained during the steady state period.

Repeat one of the runs raising the water rate to 2 cubic feet per hour.

DATA

1. Readings of air, ammonia, and water meters.
2. Temperature of the gas, entering the absorber.
3. Time to collect a given weight of liquor leaving the absorber (as a check on the water meter).
4. Absolute pressures at top and bottom of absorber.
5. Dimensions of the absorber.
6. Titration on liquor leaving the absorber.
7. Per cent ammonia in gas leaving the absorber (to be taken twice, only during operation at the steady state).
8. Barometric pressure.

REPORT

A. *Calculate for each run, tabulating results:*

1. Velocity of gas through absorption section.
2. $Dg/\mu = Du\rho/\mu$, gas through the absorption section.
3. Water rate, (weight)/(unit time)(unit area of tower cross section).
4. Per cent ammonia in gas entering absorber.
5. Per cent ammonia in gas leaving absorber:
   (a) from gas analysis.
   (b) from titrations on liquor leaving absorber and a blank on water entering absorber.
6. Partial pressures of ammonia in each phase, at top and bottom of absorber.
7. Log mean Δp for ammonia (Δp_{Lm}).
8. Average partial pressures of air over the gas film, p_{BM}.[10]

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[10] See Footnote, Experiment No 43, Calculations,

9. Mols of ammonia absorbed per hour.
10. Interfacial area of absorber.
11. Average molecular weight of air.
12. K_g.
13. k_g.
14. $\mu/\rho b_g$ for the system.
15. H.T.U. (5) for wetted wall towers, from data obtained.[11]
16. H.T.U. from the proposed equation for wetted wall towers, $= 10.9 (\mu/\rho b_g)^{2/3}(Dg/\mu)^{0.2}$. (5)

B. *Plot:*

1. $1/k$ vs $1/g$, log-log.
2. $(D/B_x)/(\mu/\rho b_g)^{0.44}$ vs $Du\rho/\mu$, log-log.

C. *Discussion:*

1. Does the plot of $1/k_g$ vs $1/g$ demonstrate that, for this system, the gas film completely controls? Determine an expression for k_g in terms of g.
2. Does water velocity affect the rate of absorption? Why?
3. Under what conditions might plot 2 be utilized to permit calculations of overall absorption coefficients?

EXPERIMENT NO. 47

Moderately Soluble Solute Gases

Object. 1. To determine the effect of gas and liquor rates upon the coefficients of absorption, using a moderately soluble gas.

2. To calculate the H.T.U. for this system.

Equipment. Same as Experiment No. 46.

Procedure. Sulfur dioxide shall be used to replace the ammonia of Experiment No. 46. Water shall be the absorbing liquid. Gas and liquor flows shall be the same as specified in Experiment No. 46. Five per cent SO_2 in air mixtures are suggested.

Part a: See Experiment No. 46, omitting the run at a water rate of 2 cubic feet per hour.

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[11] In making this calculation refer to the remarks on the calculation of N_{og}, pages 317-318. It is suggested, in a large majority of the runs, that Henry's Law may be assumed to apply, that G_M/L_M should be essentially constant, and that the ratio $(1-x)_i/(1-y)_i$ should be nearly unity. If all these conditions apply, it will be noted that Equation (33) applies.

Reference to recent papers by Colburn (8, 9) will afford a means of determining the *relative* importance of the film H.T.U. values, $(H.T.U.)_g$ and $(H.T.U.)_l$

Part b: Choose one of the Reynolds Numbers employed for gas flow rate in Part a. Repeat this run at water rates of 1.5, 2.0 and 2.5 cubic feet per hour.

DATA

See Experiment No. 46.

REPORT

A. *Calculations:*

See Experiment No. 46 omitting 13 and 16.[12]

B. *Plot:*

1. $1/K_g$ vs $1/g^n$, coordinate paper, for constant water rate runs. Assume successive values of n until the line becomes straight. From this, determine values of k_g, the relation for k_g in terms of g, and values of k_L.
2. k_g vs g, log-log, checking the accuracy of n obtained in plot 1.
3. k_L (at constant gas rate) vs water velocity.

C. *Discussion:*

1. From a value of k_g obtained for ammonia in Experiment No. 46, predict values of k_g for SO_2 at the gas rates employed in this experiment. Compare with the values of k_g obtained from plot 1.
2. Are the values of k_L at one water rate applicable to runs at a different rate? Why?

PACKED COLUMN PERFORMANCE

EXPERIMENT NO. 48

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[12] See footnote on Experiment 46. Note that Henry's Law does not apply in this case, requiring that Equation (21a) be integrated graphically for rigorous calculations. The only difficulty which should arise in this integration is determination of the term $(1 - y)_f$. Values of this term may be readily calculated when it is noted to be defined as the log mean of $(1 - y)$ and $(1 - y_e)$. Since, for most cases involved, these terms will be nearly equal, it is probable that an arithmetic mean may offer sufficient accuracy.

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Pressure Drop and Flooding Rates in Packed Columns

Object. 1. To study the pressure drop in a packed column, with packing dry.

2. To study the pressure drop in a packed column at varying rates of liquid circulation.

3. To determine the flooding gas rates in a packed column, with known rates of water circulation.

Equipment. The design features of an apparatus for such work are shown schematically in Figure 3. Employ a 6 inch tower (4 inch an absolute minimum), packed with ½ inch Berl saddles or Raschig rings. The tower may advantageously be constructed of Pyrex glass piping since this affords visual inspection of its operation under various conditions.

Two manometers are indicated, in Figure 3, for the measurement of pressure drop through the packing. This is essential, for the Δp at low flows with dry packing, and for high flows with water circulating will be so widely separated as to make the recording of both on a manometer of reasonable length impossible. Even with dry packing, for a column 6 inches in diameter with a 5 foot length packed with ½ inch Berl saddles, the variation of Δp is considerable:

$Re' = 50$; $\Delta p = 0.08''$ H_2O (approximately)
$Re' = 1000$; $\Delta p = 10.0''$ H_2O (approximately)
(air flow required = 45 C.F.M., approximately)

Procedure. The system to be employed shall be that of air flowing upward against a downward flowing water stream. Three series of runs are to be made: (a) with dry packing; (b) with a water rate of approximately 1000 pounds/(hour)(square foot of tower cross section); and (c) with a water rate of approximately 5000 pounds/(hour)(square foot of tower cross section). It is advisable to perform the run with dry packing first, since, once wet, considerable time may be consumed attempting to dry the packing completely.

Prior to the laboratory period: Obtain air and water meter calibrations. Calculate the water meter readings which must be used to obtain the required rates of water input to the column. Calculate the air meter reading corresponding to the lowest flow which may be expected to produce normal turbulence in the packed volume.

In the laboratory: **Part a:** Start the blower; adjust the air flow to the previously calculated minimum flow necessary to produce turbulent conditions. Take readings of data specified below. By experiment, obtain the highest air meter reading possible with the equipment available. Divide the range of flow into 6 equal parts; adjust the flow to these values, recording the specified data at each gas flow rate.

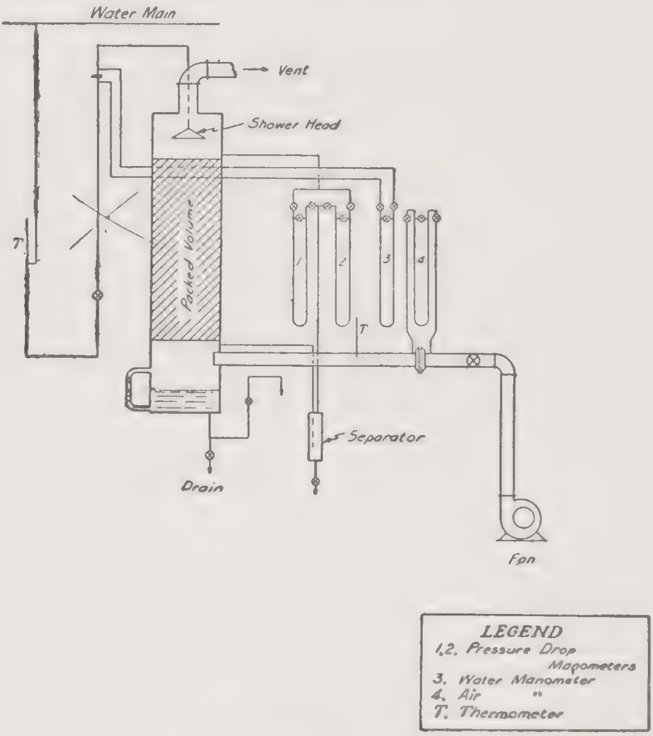

Fig. 3.—Packed Tower Layout.

Repeat this series of runs at each of the specified rates of water circulation. In these cases the maximum allowable flow shall be that at which the column tends to "flood," or fill with water.

Continue the runs made with water circulating until the column definitely floods.

Part b: Repeat the entire series of runs of Part a with a different type of packing. Do not use a packing of greater nominal size than ½ inch. Smaller sizes of packing are acceptable.

DATA

1. Diameter of tower; length of packed section.
2. Nominal size and character of packing.
3. Water and air meter readings.
4. Pressure drop manometer reading, and pressure at top of column.
5. Temperatures of air and water.
6. Time to collect a given weight of water leaving the tower (as a check on the water meter).

REPORT

A. *Calculate:* (proper items to be selected according to parts of experiment performed).

1. Air velocity, u_o, based on cross section of the empty column.
2. μ, for air (23).
3. ρ, for air.
4. Modified Reynolds Number, $d_p u_o \rho / \mu$ (33).
5. Δp, from the observed manometer reading.
6. f^* and d_p/F_A (33), for series of runs with dry packing, calculated from the observed Δp.
7. Water rate, gallons per minute.
8. Water rate, pounds/(hour)(square foot of empty tower cross section).

B. *Plot:*

1. Re' vs f^* observed, log-log. On the same sheet indicate the line of Chilton and Colburn as recalculated by White (33).
2. Δp vs u_o, each line on the plot representing the results at one constant rate of water circulation.
3. $\sqrt{u_0}$ vs $\sqrt{L/S\rho}$ at the flooding points (12).

C. *Discussion:*

1. Compare your pressure drop measurements with the equations proposed by Mach.[13]
2. From plot No. 2, estimate the loading and flooding velocities for each rate of water circulation. Compare these values with the curves of the average results of previous investigators (12, 33).
3. Explain the meaning of the terms "loading" and "flooding" velocities. Describe the performance of the column, as noted visually, at these points.

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[13] For these equations, see Badger and McCabe, *Elements of Chemical Engineering*, 2d Ed., Equations (213), (214), (215), (216), pp. 379, 380.

4. How do you explain the effect of water rate on pressure drop at constant gas velocity? What conclusions do you draw concerning the effect of gas velocity upon water holdup (quantity of water contained in the tower at any time)?
5. Predict the rate of water circulation which would flood a tower 2 feet in diameter, with a 12 foot section packed with ½-inch Raschig rings, through which 300 C.F.M. of air were being blown. Predict the pressure drop through the tower if the actual water rate is 50 per cent of the flooding rate (26, 33).

GAS ABSORPTION IN PACKED TOWERS

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EXPERIMENT NO. 49

Effect of Varying Gas and Liquid Rates on Absorption Characteristics

Object. To study the effect of rates of gas and liquor flows upon the performance of packed absorption towers.

Equipment. Figure 3 includes a large proportion of the essentials for the equipment to be employed in this experiment. A source of solute gas must be added with an orifice (or other metering device) for the measurement of rate of input. As in Figure 1, this must be introduced into the air stream after the orifice in the air line, and satisfactory mixing of the two gases must be provided before introduction to the tower. Manometers 1 and 2, of Figure 3, should be converted to read absolute pressures at top and bottom of the packed section. Gas sample tubes are to be installed in inlet and outlet gas lines. In other respects the equipment for the two experiments are identical. The tower diameter is to be 6 inches, the packing ½ inch Raschig rings, or ½ inch Berl saddles.

Procedure. Using the specified ½ inch packing, 25 C.F.M. of air will produce a value of $Re' = 500$, approximately, thus allowing a 10:1 variation of gas rate within the turbulent range. The experiment may, however, be performed if only half of this air capacity is available. Water will act as the absorbing liquid, ammonia being the solute. All runs shall be conducted with 3 per cent ammonia in the inlet gas.

A series of runs are to be made at values of $Re' = 500$, 250, 175, 125, and 90, for the gas phase, the first being omitted if sufficient air supply is not available. The water rate may be chosen at random, 500 pounds/(hour) (square foot of tower cross section) being the minimum, and the maxima being 1000 or 4000 pounds/(hour) (square foot) for the maximum gas flows equal to Re' of 500 and 250 respectively. These values are specified as being well below the loading and flooding points reported in the literature.

Prior to the laboratory period: From the calibration curves, make the usual calculations of the meter readings for the required air, ammonia, and water flows.

In the laboratory: **Part a:** Admit and regulate the air and water streams, regulating their flows to the specified values. Admit the ammonia, increasing its flow until the gas composition, as determined by the meter readings, is the specified 3 per cent. Drain the liquor from the collecting section, after approximately 5 minutes of operation under the conditions of the test, to as low a point as appears safe without breaking the liquor seal. (This will reduce the time required for the equipment to produce steady-state data.) Allow the liquor level in the collecting section to rise again until a convenient, predetermined level is noted on the sight glass. Adjust the outlet valve so that this level is maintained throughout the remainder of the run. When manometers and outlet liquor analysis indicate that a steady state has been reached, begin recording data. Continue the run for 20 minutes of operation in this steady state, recording the data listed below.

Part b: Take three of the gas velocities specified. At one of these, perform runs at three water rates within the range specified (e.g., at $Re' = 500$, water rates of 500, 750, and 1000 pounds/(hour) (square foot). At each of the remaining two gas rates, make runs at the maximum and minimum water rates.

DATA [14]

1. Manometer readings: air, ammonia, and water.
2. Tower specifications: diameter; packed height.
3. Packing specifications: nominal size; character; surface area per cubic foot.
4. Temperatures of water and gas streams.
5. Time to collect a given weight of exit liquor (as a check on the water meter).
6. Absolute pressures at top and bottom of the packed section.
7. Titration on exit liquor and a blank on inlet water.
8. Barometric pressure.
9. Analysis of exit gas.[14]

REPORT

A. *Calculate,* for each run, tabulating data:

1. Average linear gas velocity, u_o, and average mass velocity, $u_o\rho$.
2. Water rate, gallons per hour and pounds/(hour) (square foot of tower cross section).
3. Per cent ammonia in inlet gas.
4. Per cent ammonia in outlet gas: (a) from titrations on exit liquor; (b) from gas analysis (see Footnote on data).
5. μ, gas.
6. $Re' = d_p u_o \rho / \mu$.
7. Partial pressure of ammonia at top and bottom of tower, both phases.
8. Δp_{Lm} over packed section.
9. p_{BM} over packed section.
10. Mols of ammonia absorbed per hour.
11. Cubic feet of packed volume.
12. $K_g a$ and $k_g a$.
13. Average molecular weight of the gas stream.
14. H.T.U., calculated from experimental data.[15]
15. H.E.T.P., calculated from experimental data.

B. *Plot:*

1. $k_g a$ vs g, log-log, lines of constant water rate.
2. $k_g a$ vs water rate, lines of constant gas velocity.

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[14] To increase the reliability of the data, analyses of the gas entering and leaving the absorber may be taken, and material balances made. This materially increases the analytical labor and its inclusion or exclusion is left to individual instructors.

[15] See footnotes, page 313.

C. *Discussion:*

1. Show how the equation expressing k_ga in terms of g may be obtained from plot 1. Present this equation.
2. By calculation of the proper values, indicate whether or not the equipment was operated below the loading and flooding ranges.
3. Determine whether or not the gas film entirely controls. How do you explain the effect of water rate?

EXPERIMENT NO. 50

PERFORMANCE OF A CONTINUOUS ABSORPTION-STRIPPING UNIT

Object. 1. To study the operation of a continuous absorption-stripping unit.

2. To analyze the effect of gas and/or liquor velocity upon the absorption coefficients.

Equipment. Figure 4 is a line diagram of the Drexel Institute unit. The towers are of stoneware, 18 inches in diameter and 5–33 inch sections. The packing is stacked in layers as shown, being of 3 inch size and of cross section illustrated. The blower has a capacity of 135 C.F.M. at $\frac{3}{8}$ inch static pressure. (The flow over the packing used may be presumed to be turbulent at any air capacity in excess of 25 C.F.M.) The pumps have a maximum capacity of 4.5 gallons per minute; this will not flood the column even at the maximum air capacity.

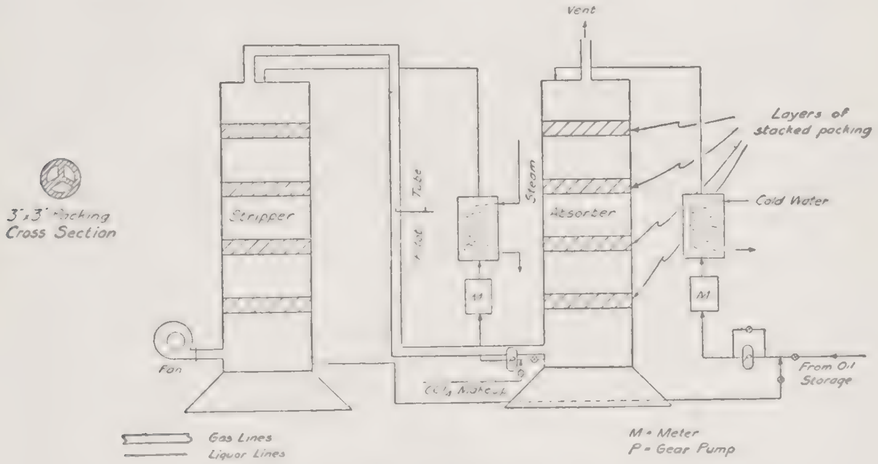

Fig. 4.—Absorber-Stripper Layout.

The advantage of such a system is the possibility of equipment of relatively large size with low solute gas requirements. The disadvantage is in the length of time required for such a system to reach the steady state, reported to be not less than two hours.

The author's recommendations for a similar equipment would be to decrease the diameter to 12 inches, to employ a full packed tower, using a standard packing of no greater nominal size than one inch, and to equip the system with fans and pumps capable of producing flooding conditions. The experiment detailed below is for the equipment of Figure 4. Minor changes, mainly in conditions of test, are all that are needed if the recommended changes are made.

Procedure. Air is used as a carrier of CCl_4 vapor, this vapor being absorbed by a hydrocarbon oil in one tower and stripped from the oil by air in the other.

Using the maximum oil flow, runs in the equipment illustrated are to be made at several gas velocities in the range of turbulence (e.g., at 30, 70, 100, and 135 C.F.M.). One of the runs is to be repeated, the oil flow being decreased to half of the pump capacity. The oil is to enter the absorber at 80 degrees F and the stripper at 115 degrees F. The original CCl_4 content of the oil shall be 5 per cent, on a weight basis.

Part a: Prepare the oil-CCl_4 mixture of specified composition. Pump the mixture through the towers at the required rate, adjusting the steam to the heater so that the temperature of the oil entering the stripper is approximately that specified. Start the blower immediately, adjusting the flow, roughly, and admitting cooling water to the cooler as soon as hot oil reaches this unit. Add CCl_4 continuously to the oil leaving the absorber to account for small losses in the gas leaving this unit. With the system thus in operation, make final adjustments of the controls to provide the specified conditions. It will be necessary to allow a period of approximately 2 hours for the attainment of the steady state. The run shall then be of 30 minutes duration.

Repeat this run at two different gas rates.

Part b: Repeat one of the runs of Part a, operating the liquor pumps at one half capacity.

Analytical: The liquid phase may be readily analyzed by the use of the Westphal balance, or the refractometer. The Edwards' gas density balance is recommended for analysis of the gas phase.

DATA

1. Meter readings for oil flows: change of reading over a time interval.
2. Pitot tube readings for air flow.
3. Tower and packing specifications.
4. Temperatures of the two phases: each tower.
5. Absolute pressures at inlet and outlet: each tower.
6. Analysis of gas phase leaving each tower.

7. Analysis of liquid phase at entrance and exit: each tower.
8. Barometric pressure.
9. Auxiliary data: angle of refraction and density vs. liquid composition; gas density vs. composition; heat capacity of oil; equilibrium oil-CCl_4 vs air-CCl_4 data.

REPORT

A. *Calculate*, for each run, tabulating results:

1. Average linear gas velocity, u_o.
2. μ, gas.
3. $Re' = d_p u_o \rho / \mu$.
4. Rate of oil circulation, L/S.
5. Mols of CCl_4 absorbed per hour, calculated from analysis of each phase.
6. Cubic feet of packed surface.
7. $k_g a$ (neglecting absorption in unpacked volume, due to low surface of contact), calculated from Plots 1 and 2.
8. H.T.U., for absorber.

B. *Plot:*

1. X vs Y diagram. Include operating line and points indicating condition at top and bottom of the absorber.
2. $1/(Y - Y_e)$ vs Y diagram for conditions of operation observed.

C. *Discussion:*

1. Is it reasonable to assume that the gas film controls? Explain.

ACKNOWLEDGMENT

This chapter is based in part upon a report presented to the Chemical Engineering Division of the Society for the Promotion of Engineering Education in June, 1936. Experimental laboratory material from the following institutions, contributed to the Chemical Engineering Laboratory Committee of the Society for the purpose of the investigation which resulted in this report, was of material assistance in its compilation: Armour Institute of Technology, Carnegie Institute of Technology, Case School of Applied Science, Cornell University, Drexel Institute, Princeton University, University of Illinois, University of Minnesota, University of Washington, University of Wisconsin, and Yale University. The authors gratefully acknowledge the assistance of these institutions and express their appreciation to the Chemical Engineering Division of the Society for the Promotion of Engineering Education for the release of the report for publication.

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Chapter IX

FILTRATION

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The testing of filtration characteristics for industrial applications is, in general, carried out to (a) design a unit when none exists, (b) outline specifications for a new unit to increase capacity when there are units already in operation, (c) determine changes in operating conditions for units already on hand.

The best specifications cannot ordinarily be attained by considering the filtration operations alone. Attention must be given to the preparation of the slurry to be filtered and to the subsequent processing of filter cake, filtrate, and wash water. The problem of consistently preparing a slurry with the most desirable characteristics is often a serious one. However, given a slurry and established characteristics of filtration products (moisture content of cake, dissolved solids left in the cake, and clarity of the filtrate), the filtration operation may be considered by itself.

The final specifications will include part or all of the following: (a) type or design of filter, (b) filtration area, (c) pressure or pressure-time relationship, (d) temperature, (e) filtration time, (f) washing time, and (g) drying time. Descriptions of filters are found in textbooks (1, 2) and handbooks (3), and will not be repeated here. Pressure filters, both *plate and frame* and *leaf* type, and vacuum filters of the *leaf* and *rotary* types will be considered.

The choice of a filtration unit is governed by the scale of operations, the resistance of the filter cake to the flow of filtrate, the washing requirements, the per cent filterable solids in the slurry, the final moisture content of the cake, and the volatility of the filtrate. Batch or intermittent units serve for small scale operations, while continuous vacuum units are well suited for larger installations, unless excluded because of high cake resistance, volatile filtrate, or special washing requirements.

FILTRATION THEORY

Batch Filter. The solid particles suspended in a slurry are separated from the liquid at the start of a filtration because they cannot pass through the meshes in the filter cloth.[1] If the mesh is not fine enough to hold back all the particles, some may pass through and give an initially cloudy filtrate. The solids held on the cloth soon become the filtering medium for the rest of the slurry. The filtrate is forced through this cake, the filter cloth, and the press channels by the difference in pressure between the inlet slurry line and the filtrate outlet line. In the majority of filtering operations the cake soon becomes the major resistance to the flow of liquid. The equivalent diameter of the individual passages through the cake is so small that the Reynolds number is always less than the critical value and the flow is in the viscous range. Poiseuille's law indicates that for this type of flow, the rate will be proportional to the first power of the pressure drop across the cake if the capillary dimensions are constant. The total rate of flow will also vary directly as the filtering area and inversely as the thickness of cake and viscosity of the filtrate. That is

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[1] Fine mesh wire screens and other porous membranes are often used instead of cloth.

$$\frac{dW}{d\theta} = \frac{1}{r}\,\frac{A(P - P_1)}{\mu L} = \frac{A^2(P - P_1)}{r\mu v W} \tag{1}$$

where $1/r$ is a proportionality constant. All actual filter cakes are compressed to some extent if the pressure on them is increased and this reduces the rate of flow for a given pressure drop by decreasing the size of the capillaries. This effect of pressure may be introduced into equation (1) by making r a function of the overall pressure drop. This procedure is valid in most cases although it is, in fact, the pressure drop across the cake alone which acts to compress the latter. It is so because cakes which are easily compressed usually offer a high resistance to the flow of filtrate and consequently the resistance of the filter cloth itself is of negligible importance. Cakes of a more granular nature are compressed much less by increase in filtration pressure and the resistance to flow of filtrate through this type of cake is lower. It follows that the resistance of the cloth may in such cases be an appreciable part of the overall resistance, but these cakes are much less sensitive to pressure changes and, therefore, larger errors in estimating the pressure acting to compress them may be tolerated.

The flow through the cloth and press channels may be assumed to follow the relation

$$\frac{dW}{d\theta} = \frac{AP_1}{b\mu} = \frac{P_1}{\text{resistance of cloth and press channels}} \tag{2}$$

Adding $P - P_1$ and P_1 from equations (1) and (2) and rearranging

$$\frac{dW}{d\theta} = \frac{P}{\dfrac{\mu r v W}{A^2} + \dfrac{\mu b}{A}} \tag{3}$$

At constant pressure this becomes on integration

$$\left(\frac{A}{W}\right)P\theta = \frac{\mu r v}{2}\left(\frac{W}{A}\right) + \mu b \tag{4}$$

This equation has been derived before in different forms (5, 4, 1). The form proposed by Ruth can be derived by rearranging equation (4) to give

$$\left(\frac{W}{A} + \frac{b}{rv}\right)^2 = \frac{2P}{\mu r v}\left(\theta + \frac{b^2\mu}{2Prv}\right) \tag{5}$$

The equivalent values of Ruth's constants C', K', and θ_o in the equation

$$\left(\frac{W}{A} + C'\right)^2 = K'(\theta + \theta_o) \tag{5a}$$

are evident. When the resistance of the filter medium is negligible $b = o$ and both forms reduce to

$$\left(\frac{W}{A}\right)^2 = \left(\frac{2P}{\mu r v}\right)\theta \tag{6}$$

The factors r and b are functions of pressure and for engineering calculations the relations $r = r'p^s$ and $b = b'p^m$ are recommended.

Ruth's equation (5a) when differentiated and rearranged gives

$$\frac{d\theta}{d(W/A)} = \frac{2}{K'}\left(\frac{W}{A}\right) + \frac{2C'}{K'}. \tag{6a}$$

This equation indicates that the reciprocal of the rate of filtration per unit area is linear in the weight of filtrate per unit area. A graph of these two variables provides a convenient means for evaluating the constants C' and K' since the slope of the straight line is $2/K'$ and the intercept on the ordinate is $2C'/K'$. The reciprocal rate, $d\theta/d(W/A)$, in this special case may be readily obtained from a tabulation of the filtration data as (W/A) and corresponding values of the total filtering time θ. If $\Delta(W/A)$ is the difference between any two values of (W/A) in the table and $\Delta\theta$, the difference between the corresponding values of θ, then the ratio $\Delta\theta/\Delta(W/A)$ is equal to the derivative $d\theta/d(W/A)$ at a value of (W/A), half-way between the two values in the table or at $(W/A) + \frac{1}{2}\Delta(W/A)$. A sample tabulation using total filtrate volume V instead of weight per unit area is given on page 362.

A complete mathematical presentation of the theory of filtration, and the derivation of the Ruth equation are to be found in reference 4. Later papers by Ruth and by Weber elaborate on the application of this equation. It is derived by the same fundamental considerations as the earlier one of Lewis and Almy, but is to be preferred in some cases because it permits making corrections for errors in determining zero filtering time.

It is not possible to calculate filter performance without some experimental data. Experience with these equations, as applied to experimental filtration data, indicates that they can be applied in computing filter requirements when data are available on the filtration characteristics of a certain slurry at the chosen operating pressure.

Washing. Dissolved material in the filtrate held in the pores of the cake often requires that the latter be washed. In the plate and frame type filter, when the frames are filled solidly (3), the wash passes through half the cake in a direction opposite to that of the filtrate. For other filtering operations, the wash passes in the same direction as the filtrate and probably follows the same paths through the cake if air has not been allowed to enter between the filtering and washing stages. The first part of this type of washing operation is known as displacement washing (2). The filtrate in the solid is displaced by the incoming wash and the concentration of dissolved material in the effluent does not decrease appreciably. When the wash breaks through the cake, the concentration in the effluent then falls rapidly and experiments (7) indicate that its concentration is proportional to the concentration of dissolved material still in the cake. Such a relation results in the semi-logarithmic equation

$$\ln c = -\frac{k'w}{vW} + \ln c_o \tag{7}$$

where the constants k'/v and c_o are the slope and intercept on a graph of ln c versus w/W (ratio of wash water to filtrate). This equation may not hold when washing plate and frame presses with solid filling the frames, or when air is allowed to enter the cake, but the method of plotting is convenient. These data are useful in determining the amount of washing that is economically feasible.

Air Drying. A part of the wash water or filtrate remaining in the cake at the end of the washing or filtering parts of the cycle may be removed by an "air dry." Although the moisture content is considerably reduced by passing air through the cake in this stage, a heat balance readily shows that the major part of this reduction is by a displacement or entrainment process and not by evaporation. Data on the moisture content of the cake in relation to the amount of air passed through are of importance.

CONTINUOUS ROTARY DRUM FILTERS

On continuous rotary drum filters, the filtering, washing, air drying, and cake discharge stages of the cycle are carried out in a continuous sequence. The relative time allotted to the first three of these stages is controlled by varying (a) the per cent of the drum surface area which is submerged in the slurry, (b) the position of the wash water sprays or weir, (c) the volume of the wash water, and (d) the setting of the automatic suction valve ports. The cake thickness and the rate of filtration for a given slurry will depend on the pressure drop and speed of rotation of the drum in addition to the fraction of the drum surface submerged in the slurry trough. The ease with which the cake will discharge from the cloth is of importance. Any cake left in the pores of the cloth or as a layer of undischarged cake will act as additional cloth resistance.

A discussion of the application of the Ruth equation to continuous rotary drum filters is found in reference (6). A brief presentation of the general theory is made herewith:

Filtration Stage. Since the pressure is constant during the filtration stage, equation (4) will apply. During each revolution any point on the drum surface is filtering for ψ/N units of time. Therefore, in the above equation $\theta = \psi/N$ and $W = W_r$ or

$$\left(\frac{A}{W_rN}\right)P\psi = \frac{\mu rv}{2}\left(\frac{W_r}{A}\right) + \mu b \tag{8}$$

where W_rN/A = weight of filtrate per unit time per unit area. It will be noted that a graph of $(A/W_rN)P\psi$ versus W_r/A is linear according to this equation if b is constant and it has an intercept on the $(A/W_rN)P\psi$ axis of (μb). Reference to equation (2) will show that this intercept is the cloth resistance per unit area. In this instance it will include the resistance of any undischarged cake.

Washing Stage. The wash water is sprayed or run from a weir on to the surface of the filter cake. It must not disintegrate the cake or flow down into the slurry tank. Consequently, air is usually sucked through the cake after it leaves the filtering stage, and a mixture of air and wash water are drawn through the cake during washing. These conditions are difficult to reproduce in small scale, batch, leaf washing tests. When washing is important, the characteristics of the filter in this stage make up a part of the information which can be obtained by actual operation of a rotary filter. A preliminary estimate of the fraction of the drum surface to be used for washing, ψ_w can be obtained by analogy to submerged leaf washing.

From equation (3), neglecting cloth resistance, and calling the washing rate equal to the filtering rate at the end of the filtering stage, the washing time per revolution is

$$\frac{\psi_w}{N} = \frac{w_r/A}{\dfrac{d(w/A)}{d\theta}} = \frac{(w_r/A)\mu rv(W_r/A)}{P} = \frac{w}{W}\,\frac{\mu rv(W_r/A)^2}{P}$$

and

$$\psi_w = \frac{\mu rv}{PN}\left(\frac{W_rN}{A}\right)^2\left(\frac{w}{W}\right)$$

inserting W_rN/A from equation (8) and again neglecting cloth resistance

$$\frac{\psi_w}{\psi} = 2\frac{w}{W} \qquad (9)^1$$

This equation indicates that for a washing rate equal to that of a submerged leaf, the ratio of the fraction of the surface of the drum washing to the fraction of the surface filtering should be twice the ratio of wash water to filtrate. Actually, it will be necessary to use a greater fraction of the surface than this for washing, to give the required ratio of wash water to filtrate.

Air Drying Stage. This stage on the continuous filter is similar to that on the batch leaf filter and the characteristics of the former should correlate with those of the latter. The vacuum pump used to suck air through the cake during the air drying stage consumes the major fraction of the power used to operate the filter. Therefore, a knowledge of the relationship between the final moisture content of the cake and the weight of air passed through is important. It may be estimated by experiments with a single leaf filter. If w_a/W is the ratio of weight of air used to weight of filtrate collected which will yield a cake with a given moisture content, the relation between this ratio and the ratio of air drying area to filtering area may be derived as follows: (10). Assume that the rate of air flow through the cake can be approximated by an equation of the same form as that for the liquid flow. Neglecting cloth resistance

$$\frac{dw_a}{d\theta} = \frac{PA^2}{\mu_a kvW_r} \qquad (10)$$

and

$$\frac{\psi_a}{N} = \frac{w_{ar}\mu_a kvW_r}{PA^2} = \left(\frac{w_a}{W}\right)\frac{\mu_a kv}{PN^2}\left(\frac{W_rN}{A}\right)^2$$

or substituting for $(W_rN/A)^2$ from equation (8) and rearranging

$$\frac{\psi_a}{\psi} = 2\left(\frac{w_a}{W}\right)\left(\frac{\mu_a}{\mu}\right)\frac{k}{r} \qquad (11)$$

The value of k may be determined on a leaf filter by experiments on the flow of air through a freshly formed wet cake, applying equation 10. The factor k varies with the moisture content as well as with the pressure and deviations may be expected when large percentages of moisture are removed in air drying.

When k is small, large volumes of air may be passed through the cake without excessive power costs. It is often advantageous under these cir-

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[1] This equation neglects the ratio of the viscosity of the wash water to that of the filtrate which is only justified for values of this ratio near unity.

cumstances to preheat the air and increase the amount of evaporation. The intimate contact allows the air to approach equilibrium with the cake and results in good heat economy.

The cake volume may be determined by measurement of its thickness and area. When employing this method, the area should be subdivided into a number of equal parts and the thickness of each part measured. These values can then be averaged. It is sometimes more convenient to obtain this volume from analytical data on the slurry, filtrate, and wet unwashed cake. Thus, by taking the volumes of insoluble solid and filtrate as additive and setting up a material balance around the filter, it can be shown that when

d_s = specific gravity of slurry
d_f = specific gravity of filtrate
f = weight fraction of solvent in the filtrate
f_s = weight fraction of solvent in the slurry
f_c = weight fraction of solvent in wet unwashed cake

the volume of insoluble solid in unit weight of slurry is

$$\left(\frac{1}{d_s} - \frac{f_s}{d_f f}\right)$$

cc./gm. while the weight of this solid in gm./gm. is $(1 - f_s/f)$. The ratio of cu. in. of cake to lbs. of filtrate, v, then becomes

$$27.7\left(\frac{f - f_c}{d_s(f_s - f_c)} - \frac{1}{d_f}\right)$$

if the voids in the sample of cake analyzed were filled with filtrate solution.

The specific gravity of filtrate and slurry may be determined with a hydrometer or a Westphal balance. If the slurry tends to settle rapidly, a pipetted volume should be weighed on an analytical balance to secure the proper precision.

The weight fraction of solvent in the filtrate, slurry, and cake are obtained by evaporating the samples on a steam bath and then drying to constant weight in an oven at 110° C. Some precipitates dehydrate to varying degrees and special care must be taken to check results.

A thin steel ruler is convenient for measuring cake thickness.

PREPARATION OF SLURRY

Much of the difficulty encountered in obtaining reproducible results from filtration tests lies in the slurry. The suspended particles themselves and the resistance they offer in the filter cake are usually very sensitive to the conditions of formation, aging, and the method of handling the slurry. For this reason, a slurry made up by precipitation, kept agitated by a high speed stirrer and pumped to the press by a centrifugal pump may change rapidly with time and give inconsistent test data. A better practice is to prepare the slurry some time before testing and to use low speed, paddle type agitators and a monte-jus or a low speed, rotary, positive displacement pump for obtaining the required pressure and maintaining circulation. In plant testing, the identical slurry that is to be filtered in the plant size equipment should be charged to the test filters, if possible. Otherwise, the plant method of preparation and handling should be followed closely, if the results are to be significant. Slurries which have been used by various investigators are described below.

"Hyflo Super-Cel" (Johns Manville Company) has a very high porosity and gives excessively high rates of filtration. For this reason, it is not satisfactory for laboratory testing. The ordinary Filter-Cel suspended in water gives lower rates of filtration, but the cake resistance increases on standing and results are not consistent. However, if the suspension is made up in 3 or 4 weight per cent salt (NaCl) solution, the cake resistance is lower but quite consistent. A mixture of 0.3 pound of dry Filter-Cel in one gallon of solution is suggested. The Filter-Cel as received contains considerable water and must be dried and analyzed if the exact composition of the slurry is to be determined (8).

Suspensions of calcium carbonate vary widely, depending upon the method of preparation. Two types of crystals may be formed—calcite which gives crystals modelled after a rhombohedron and in the trigonal system, and aragonite which is composed of needle-like crystals in the rhombic system. The suspension may be made by adding

a. precipitated calcium carbonate to water,
b. milk of lime to a solution of sodium carbonate (soda ash),
c. a solution of calcium chloride to a solution of sodium carbonate (soda ash),
d. a solution of ammonium carbonate to a solution of calcium chloride.

Suspensions of *precipitated calcium carbonate*, which can be bought dry, by the barrel, have been used (9) and found to be inexpensive, easily handled, non-hygroscopic, and fairly incompressible. The slurry concentration ranged from 5 to 11 per cent solids by weight and gave reproducible results which correlated. It is wise not to use over 15 per cent by weight.

When a precipitate is formed by *liming* a soda ash solution, the original particles take the form of the lime particles and on continued digestion at elevated temperatures for 6 to 8 hours gradually change over to calcite. These changes in crystal structure cause corresponding changes in cake resistance. Although such a slurry may be stabilized by washing it free of caustic and allowing sufficient time between preparation and use, the procedure is lengthy. The cake will have appreciable resistance and a fair degree of compressibility. A suspension of *calcite* is formed when a hot (85° C.) solution of calcium chloride is added slowly to a hot (85° C.) solution of soda ash with vigorous agitation. A slight excess of sodium carbonate should be used. The suspension is digested at 80° C. for two hours, diluted with cold water, and cooled to 25° C. The relative proportions are indicated as: 7 gallons of water plus sufficient soda ash to contain 10 pounds of sodium carbonate and for the other solution 5 gallons of water plus calcium chloride equivalent to the sodium carbonate (usually about 15 pounds of technical grade). The resulting slurry is diluted to 30 gallons. The cake resistance is relatively low and not entirely reproducible. If 0.1 per cent urea is present in the calcium chloride solution, the above procedure gives aragonite crystals and the filtration characteristics are distinctly altered (8). A calcite slurry composed of larger crystals, which is more suitable for filtration experiments, may be prepared by adding a solution of ammonium carbonate at room temperature to a hot (85° C.) solution of technical grade calcium chloride. Rapid mixing may be secured and foaming minimized by introducing the solution to the recirculation line just before it enters the pump.

Ferric hydroxide slurry may be prepared by mixing a solution of ferric chloride and ammonium hydroxide, using a slight excess of ammonium hydroxide and then gently boiling until the excess of ammonium hydroxide has evaporated. The precipitate settles completely in the quiet solution. Remove the mother liquor by decantation and wash three times with hot water by decantation. A three per cent suspension of this ferric hydroxide in water is suitable for filtration.

A nonhomogeneous slurry of aluminum hydroxide and filter aid, which gives a cake of medium resistance and compressibility, is produced by adding soda ash solution to aluminum sulfate solution until a *p*H near the isoelectric point of the hydroxide is reached. The solutions may be made up in the proportions of 20 pounds of technical aluminum sulfate to 100 pounds of water and 9 pounds of soda ash to 100 pounds of water. Add the soda ash solution slowly with vigorous agitation until the green end point is reached, using methyl red-methylene blue indicator. After the reaction is complete, add one pound of Celite 110 (Johns Manville Company) and if washing experiments are to be performed on the cake, the addition of one pound of salt will simplify analysis of the wash water effluent.

The cakes formed from relatively granular materials like calcium carbonate may be repulped and returned to the slurry tank. The ferric and aluminum hydroxides do not repulp readily.

NOMENCLATURE (Basic Units: In., Lbs., Min.)

A = total filtering area, sq. in.
b = a constant in equation for flow through cloth and press channels $(\text{min.})^2(\text{in.})/\text{lbs.}$
b' = coefficient in equation $b = b'(P)^m$
c = concentration of dissolved solids in wash effluent, lbs./cu. in.
c_o = constant of integration, intercept at $w/W = 0$
ψ = fraction of total filtering surface submerged in slurry at any instant
ψ_w = fraction of total filtering surface exposed to washing at any instant
ψ_a = fraction of total filtering surface exposed to air drying at any instant
k = proportionality constant in air drying equation, min./lb.^{2}
k' = constant in washing equation, cu. in./lb.
L = thickness of cake at time θ; $L = vW/A$
m = exponent
N = revolutions per minute of rotary filter
P = total pressure drop across cake, cloth, and press, lbs./sq. in.
P_1 = pressure drop through filter cloth and press channels
r = a constant in equation for flow through cake, min.^{2}/lbs.
r' = coefficient in equation $r = r'P^s$
s = exponent of compressibility
v = ratio: volume of cake to weight of filtrate, cu. in./lb.
W = weight of filtrate obtained up to time θ, lbs.
W_o = weight of filtrate in plate and filtrate lines
W_r = weight of filtrate per revolution, lbs.
w = weight of wash at time θ, lbs.
w_r = weight of wash per revolution
w_a = weight of air sucked through cake in air drying at time θ
w_{ar} = weight of air per revolution
θ = time, min.
μ = viscosity lbs./min. × in. (multiply viscosity in centipoises by 0.00336 to convert to lbs./min. × in.)
μ_a = viscosity of air
ρ = density of filtrate

Equipment. The design of filters and details of operation will vary with the results desired and valuable experience may be derived from testing very simple as well as rather costly filtration units, if quantitative data are recorded and these are explained in the light of present theory. For this reason, no attempt will be made to suggest standard units. For general experimental purposes, however, a vacuum leaf is the least expensive and can be used to give interesting and important information. A pressure leaf filter, with a single leaf, is also convenient for experimental work. It should connect with two pressure containers, one for slurry, one for wash water. A small plate and frame type press is desirable. The continuous rotary vacuum filter with its accessories is relatively more costly and should be considered only after the above three types are on hand. Small (25-150 sq. in.) filtration areas will give good results and, in addition, require smaller volumes of slurry and smaller storage and handling facilities. Some modifications in the usual commercial type filters are usually advantageous to improve the accuracy of measurement of pressure drop, time, and filtrate volume and to reduce the resistance of the press channels. These will be discussed in connection with the particular experiment in which the equipment is to be used.

EXPERIMENT NO. 51

FILTRATION: USING A VACUUM LEAF

Object. To determine the filtration equation with a vacuum leaf.

A filtration experiment using the simplest and least expensive equipment is a vacuum filtration using a single leaf from a pressure filter, or a specially constructed disc as the cake forming surface.

Equipment. The necessary equipment arrangement is shown in Fig. 1.

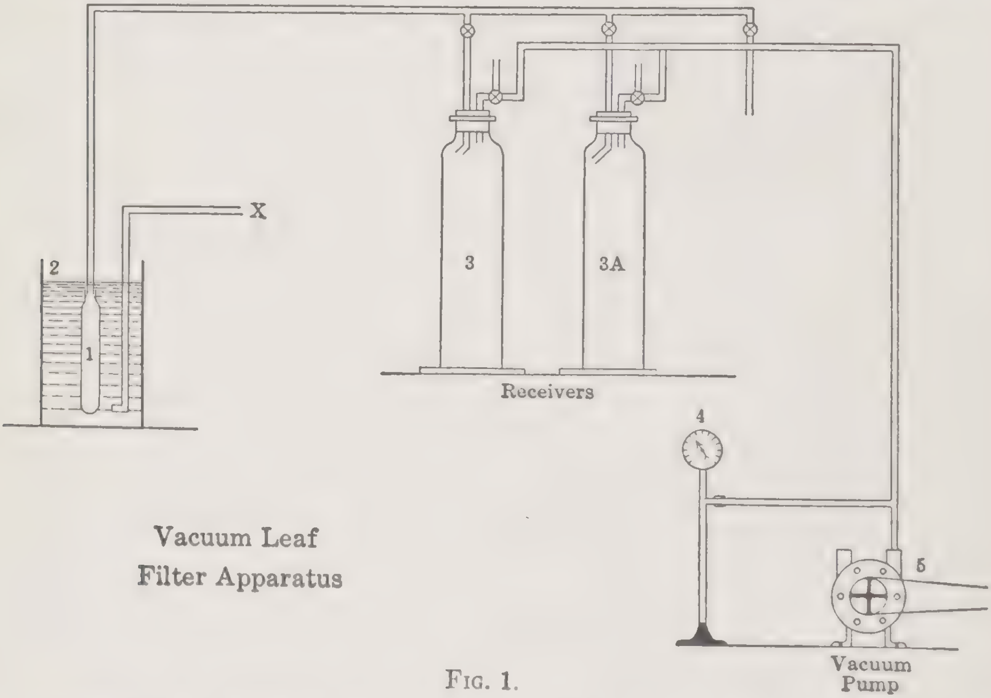

FIG. 1.

Procedure. A filter leaf (1) is immersed in a trough containing a 12% suspension of calcium carbonate in water. Admission of air through the pressure line X keeps the material in suspension.

The filter leaf connects to two graduated cylinders, 3 and 3A of 2000 m.l. capacity, through a glass tube in a two hole rubber stopper. Another glass tube connects through a rubber hose, to the vacuum gauge (4) and the vacuum pump (5). The stopcocks, at the top of the receivers, permit the use of either one at a time and while one is filling the other may be emptied.

The slurry tank (2) should have a capacity of at least four gallons.

The vacuum pump should have a capacity adequate to maintain, at least, a vacuum of 14 in. and this should be held constant, by adjusting the stopcocks, during the entire filtration cycle.

It has been found that the operating conditions likely to cause trouble are: (a) variation in vacuum, (b) variation in slurry concentration due to settling, (c) failure to accurately correlate the reading of the filtrate volume with the time, and (d) excessive resistance in filtrate lines.

Each experimenter must rely on his own judgment and ingenuity to overcome these difficulties.

Repeat the filtration of the same slurry three times, with the same operating conditions.

REPORT

Tabulate your data under the headings:

Time	Filtrate Volume	Difference in Volume		
θ	V	ΔV	$V + \dfrac{\Delta V}{2}$	$\dfrac{\Delta\theta}{\Delta V}$

Your data as obtained is a time-volume relationship. A plot of total volume against time will give a parabola which does not readily lend itself to calculation. An indirect method, originally given by Ruth, (4), follows from equation (5*a*) above. That this equation also applies when the total volume of filtrate, $V = W\rho$, is measured instead of its weight, W, is evident if both sides are multiplied by ρ^2A^2. Equation (5*a*) then becomes $(W\rho + C'\rho A)^2 = K'\rho^2A^2(\theta + \theta_0)$, or $(V + C)^2 = K(\theta + \theta_0)$, which can be used in the same way as equation (6*a*). Thus a graph of $\Delta\theta/\Delta V$ versus $V + \Delta V/2$ gives a straight line according to the equation

$$\frac{\Delta\theta}{\Delta V} = \frac{2V}{K} + \frac{2C}{K}$$

in which the slope is $2/K$ and the y-intercept is $2C/K$. The x-intercept is, therefore, $\dfrac{2C}{K} \cot \tan^{-1} \dfrac{2}{K}$ or is equal to C. This linear plot offers a simple method for determining C and K.

Plot the data from the vacuum leaf, using the indirect method, and determine the values of C and K. See Experiment 52 for sample data and a tabulation. Discuss the influence of the filtration pressure on C and K.

EXPERIMENT NO. 52

Using a Single Pressure Leaf

Object. To determine the effect of pressure with a pressure leaf filter.

Equipment. A pressure leaf filter, with a single leaf. The assembled unit is shown in the sketch, Figure 2, on page 361. The separate pieces may be identified as:

(1) Pressure container for wash water.
(2) Pressure container for sludge.
(4) Pressure container for filter leaf with the filter leaf inside.
Graduated cylinder 2000 m.l. capacity used as the filtrate receiver.

Details of pipe lines and valves are shown as well as details of the construction of the filter leaf. The skeleton for the filter leaf is Monel metal throughout.

Procedure. About five liters of sludge containing 10-12% calcium carbonate in suspension is made up and put in containers 2 and 4. It has been found that "air float" calcium carbonate purchased in 400 lb. barrels is a satisfactory material for sludge making. The particle size is quite uniform so that no difficulty is encountered in obtaining concordant results in a series of filtrations. 1 lb. salt per 100 lbs. of water should be added if washing tests are to be made.

With the sludge in its container the cover is bolted on and container 1 is filled with wash water. The filter leaf, which is attached to the cover, is bolted in place and air, under the desired pressure, is admitted to container 2. The air line enters this container on the side wall near the bottom and the line is continued inside the container to a point just over the sludge outlet. This construction and method of operation assures satisfactory agitation of the sludge within its container.

The valve on the filtrate line is closed during the initial period while a petcock in the cover of the leaf container is open. The petcock is closed as soon as a small quantity of sludge runs out. The valve on the filtrate line is then opened, cake formation starts, and the timing of the filtration operation begins. Accurate coordination of filtrate volume and time

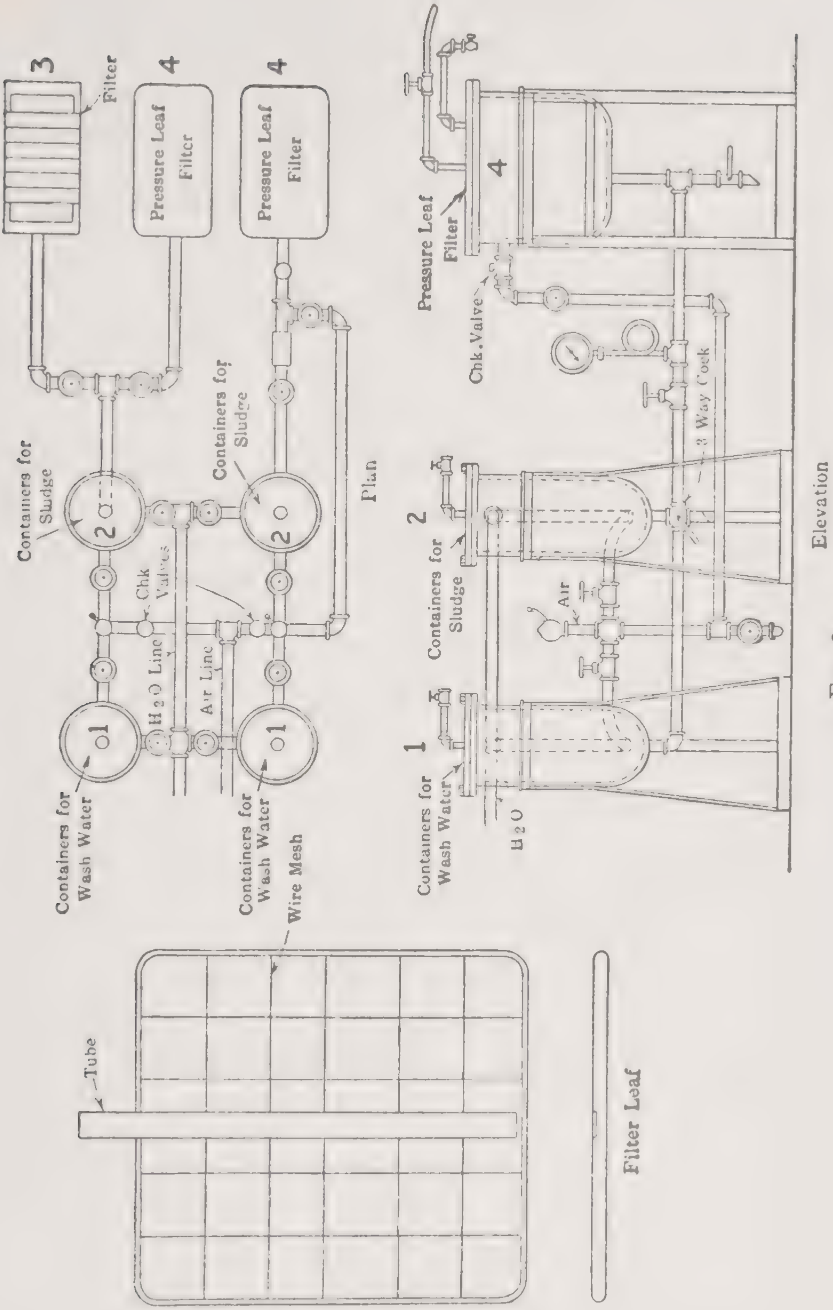
3
Filter
4
Pressure Leaf Filter
4
Pressure Leaf Filter
Containers for Sludge
2
Containers for Sludge
2
Chk Valves
H2O Line
Air Line
1
1
Containers for Wash Water
Containers for Wash Water
Plan
Wire Mesh
Tube
Filter Leaf
Pressure Leaf Filter
4
Chk. Valve
3 Way Cock
2
Containers for Sludge
Air
1
Containers for Wash Water
H2O
Elevation

Fig. 2.

is most essential in securing data which may be used satisfactorily in the calculations.

Additional agitation of the sludge may be secured, if desired, by opening a petcock on the top of the sludge container so that a small amount of air continually escapes during filtration.

The filtration is concluded by shifting the three-way cock at the base of the sludge container, closing the line from the latter and opening the line from the wash water container. This is done only when *all* the sludge has been filtered.

During the washing operation samples of wash water should be collected and analyzed for dissolved solid. At the conclusion of the washing operation, the valve at the bottom of the leaf container is left open, the petcock on the top of the sludge container is opened, the valve on the air line entering near the top of the leaf container is opened, and the air line to the bottom of the sludge container is closed. All these changes in the valve positions should be made without permitting the pressure in the leaf container to drop. As soon as all the liquid has been forced from the leaf container back into the sludge container, the petcock on top of the sludge container is closed. Air is then passed through the cake until it is dry. The dryness of the cake is indicated by the quantity of water expelled per unit of time, and when no more water is expelled by the air pressure, the experiment is concluded.

Repeat the filtration at another pressure omitting the washing operation.

SAMPLE DATA AND CALCULATIONS

PRESSURE LEAF FILTER[1]

θ (sec)	$\Delta\theta$ ~~(liters)~~	V (liters)	ΔV	$V+\Delta V/2$	$\Delta\theta/\Delta V$
0		0.00			
15	15	0.58	0.58	0.29	25.8
30	15	0.95	0.37	0.77	40.5
45	15	1.24	0.29	1.10	51.7
60	15	1.49	0.25	1.37	60.0
75	15	1.70	0.21	1.60	71.5
90	15	1.89	0.19	1.80	79.0
105	15	2.08	0.19	1.99	79.0
120	15	2.26	0.18	2.17	83.4
135	15	2.43	0.17	2.34	88.3
150	15	2.59	0.16	2.51	93.6
165	15	2.73	0.14	2.66	107.0
180	15	2.87	0.14	2.80	107.0
195	15	3.00	0.13	2.94	115.0

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[1] From report by Levy and Schreiber (Ch. E. Laboratory, Armour Institute of Technology).

Cake weight = 52.0 lb./sq. ft./hr.
Filtrate volume = 162.0 liters per sq. ft./hr.
Moisture content 23.5%
After drying 15 min. at 25 lb./sq. in. air pressure
Cake thickness, $\frac{9}{16}$ in.
Filtration pressure 25 lb./sq. in.
Sludge concentration 10% $CaCO_3$ by weight.

This data is used in solving the Ruth equation as explained in Experiment 51 and also on page 359.

EXPERIMENT NO. 53

A Simple Plate and Frame Filter

Object. To compare filtration and washing on a plate and frame filter with that on a pressure leaf as in Experiment 52.

Equipment. The equipment used is shown in Figure 2. This equipment has been found to be more satisfactory for experimental work than a larger plate and frame press.

It consists of (3) the filter press, (2) the pressure container for the sludge, (1) the pressure container for the wash water, (a) the graduated cylinder serving as a filtrate receiver.

Procedure. The operation is conducted as has been described in Experiment 52, using the same sludge concentration and the same pressures.

REPORT

The same type of data is taken and the same calculations and graph are made.

Compare the filtrate volumes per unit of time per unit of filter area.

Discuss the variations found in the data for the two filtrations.

Compare the elapsed time in a filtration cycle, starting the timing for the cycle when filtration begins and ending the timing period when the equipment is all clean and ready for a second filtration.

Plot curves of the dissolved solids content versus the volume of wash water for the two experiments and compare the washing efficiencies.

EXPERIMENT NO. 54

Filtering, Using an Industrial Type Plate and Frame Filter

Object. To study the operation of industrial plate and frame filters.

The prior filtration experiments are conducted essentially to secure preliminary engineering data for the design of larger scale equipment. As the size and design of the equipment is changed variations in the filtration characteristics are expected. This experiment is essentially an operating one to check the experimental data from industrial type filters against that from smaller scale tests.

Equipment. A plate and frame filter of the customary laboratory size. Frames should have a thickness of ¾ to 1 in. and dimensions of about 12 in. × 12 in. It is desirable to have the filter constructed for back washing.

The slurry tank must be provided with an efficient agitator. The slurry may be pumped to the filter using a piston pump or a centrifugal pump. It is convenient to have a by-pass line between the pump and the filter equipped with a relief valve set at the desired filtration pressure.

The wash water can, most conveniently, be brought in through the pump thus maintaining the same pressure during filtration and washing.

The slurry should be a 10-12% suspension of calcium carbonate in water containing about 1% of sodium chloride as the soluble constituent to be removed by washing.

Procedure. A quantity of water, estimated to be ample for the filtration period, is placed in the tank. The required amount of sodium chloride is added and the agitator started. The calcium carbonate is added slowly so that all the material remains in suspension as added. When this has been completed, start the pump and keep the relief valve on the by-pass line adjusted to secure the desired filtration pressure.

The filtrate volume per unit of time is determined by weighing. It is necessary to correlate *accurately* the time and the filtrate volume. Conclude the filtration when the frames are approximately filled. (What will be the basis for this decision?)

The wash water is then passed through the cake, taking a sample of wash water from each unit volume to be used for determining the amount of sodium chloride.

A sample of the cake is taken for the determination of moisture and dissolved solids.

PRELIMINARY REPORT

Calculate frame capacity, in terms of wet calcium carbonate, and show figures for the amount of dry calcium carbonate required in the slurry.

Calculate the size of the press channels required from the viewpoint of fluid flow. Are there any apparent restrictions to such flow?

REPORT

Tabulate time and filtrate volume.

Plot $\Delta\theta/\Delta V$ in seconds per liter against $V + \Delta V/2$ in liters and calculate C and K. Determine the moisture content of the press cake, and its soluble solids content.

EXPERIMENT NO. 55

EFFECT OF SLURRY CONCENTRATION AND WASHING CHARACTERISTICS

Object. This is a variation of the experiment on the plate and frame press to emphasize the effect of slurry concentration and supply data on cake washing.

Equipment. A plate and frame press, equipped with separate inlets for slurry and wash water, and separate outlets for filtrate and wash effluent, is suitable. Only one frame is used, followed by a plate with filtrate and wash outlet and then a blank plate. One head of the press serves as a plate for the other side of the frame and contains a filtrate outlet and a wash water inlet opening. A frame about 1 inch thick is appropriate. The other equipment may be similar to that described on page 364. The capacity of the plates and filtrate lines should be known.

The slurry to be filtered should produce a cake of reasonable resistance and may vary from aluminum hydroxide with a filter aid to rather fine grained calcium carbonate. If the latter is used in tap water for the last three runs, there should be no effect due to changing concentrations of salts in the filtrate.

Other necessary apparatus includes a hydrometer, stop watch, burette, scales, balance, 12-100 cc. beakers, 10-250 cc. Erlenmeyer flasks.

Procedure. Run 1. In a portion of prepared slurry dissolve about 1 lb. of salt per 100 lbs. of water and mix thoroughly. Inspect the press, check the valve settings, and assemble. Start the slurry through the feed lines and, with the valve in the filtrate line closed, fill the frame, venting air through the special cock placed in its top. As soon as the slurry appears, close this cock and, when the desired pressure is reached, open the filtrate line. Start the stop watch as soon as filtrate appears. Fill the frame at a pressure of 40 lbs. per sq. in., tabulating weight of filtrate against time. A sudden decrease in filtering rate will indicate that the frame is full. If any doubt exists, a rough graph of time/weight of filtrate versus weight of filtrate will show a break at this point. During the filtration, collect a sample of the slurry for moisture determination and measure its temperature.

After the filtration, shut off the valve in the slurry line and exchange the filtrate receiver for one to receive wash effluent. Two samples of the filtrate should be saved, one for titration and one for moisture determination. Open the valve in the wash water line and start washing slowly at first. The first 100 cc. should be weighed and saved for titration. Gradually increase the washing pressure to 40 lbs. per sq. in. and collect larger samples, saving 100 cc. of each and discarding the rest, until a weight of wash water equal to the weight of filtrate has been passed through. Record the weight of wash effluent against time. About 10 to 12 samples should be collected. Measure the thickness and weight of the cake.

Titrate suitable portions of each sample of wash effluent with 0.01 *N* silver nitrate solution to determine the concentration of salt. The portion should be small enough so that each titration requires no more than 30 to 40 cc. of the $AgNO_2$ solution. Dilute to 100 cc., add 1 cc. of dilute acetic acid, and 1 cc. of $KCrO_4$ indicator solution.

Run 2. Repeat the filtration part of the above run using a fresh portion of the slurry without the addition of salt. Measure the specific gravity of the slurry and filtrate and take samples for moisture determination. Do not wash or air dry the cake, but measure its thickness and weight, and take representative samples of about 25 gms. for moisture determination.

Make two additional runs identical with Run 2, except that the slurry should be diluted with 50% of its weight of water for Run 3 and with 100% for Run 4.

REPORT

Record the following measurements:

a. filtration area
b. capacity of plates and filtrate lines
c. filtration pressure

for each run:

d. description of the slurry
e. tabulation of weight of filtrate against time
f. temperature of slurry
g. specific gravity of filtrate and slurry as indicated above
h. gross, tare, and net weights of samples for moisture determination
i. weight of cake
j. average cake thickness

and for the first run:

k. tabulation of time versus weight of wash effluent
l. cc. of 0.01 *N* AgNOr solution required for titration per cc. of filtrate and each sample of wash effluent.

CALCULATIONS

From the analytical data or the measurements of cake thickness determine the value of v for each run, the per cent solids in the slurry and filtrate, and, from the latter values, the per cent insoluble solids in the slurry. Plot the filteration data as $P\theta/(W/A)$ against $(W + 2W_o)/A$,[1]

---

[1] The filtrate capacity of the plates and filtrate lines, W_o, may be taken into account by a modification of equation (4). The resistance of a unit area of the cake corresponding to W_0 lbs. of filtrate is $\mu r v \frac{W_o}{A}$. Introducing this resistance into equation (3) and integrating gives

$$\left(\frac{A}{W}\right) P\theta = \frac{\mu r v}{2}\left(\frac{W + 2W_0}{A}\right) + \mu b \qquad (4a)$$

and obtain values of r and b for each run. Show graphically the relation between r and the per cent of insoluble solids in the slurry, noting any effect of the per cent of soluble solids.

On semi-logarithmic coordinate paper plot c on the logarithmic scale against w/W on the linear scale and compare the shape of the curve with that predicted by equation (7). Explain. Determine the washing rate during the time the pressure was maintained constant and compare this with the final filtering rate.

Discuss the effect of the per cent insoluble solids in the slurry on the cake resistance, rate of flow of filtrate, and optimum thickness of cake. If the per cent soluble solids in the slurry were high, what effects would be expected on dilution?

Can any changes be suggested in the procedure of Run 1 for washing the cake? Discuss their advantages and disadvantages.

EXPERIMENT NO. 56

Filtration Using a Continuous Rotary Vacuum Filter

Object. To study slurry filtration by means of rotary vacuum filter.

Equipment. A small rotary vacuum filter of the Oliver type is satisfactory (Fig. 3). The filtration cycle and the theoretical presentation pertaining to this filtration are presented on pages 352 to 354, inclusive.

The slurry selected for filtration should be of the easily filterable kind. Corn starch is satisfactory and much corn starch is filtered industrially on vacuum filters of the continuous type.

The slurry should contain about ten per cent solids in suspension and the cake, after studying its characteristics, may be repulped and reused.

A satisfactory set-up for this experiment is shown on page 368 and the sketch, Fig. 4, depicts the filter construction.

The filter is a rotating drum, immersed to a definite depth in the slurry tank. The drum is divided, peripherally, into segments. These segments are connected, through pipe lines, to the axis which in turn, connects, through an automatic valve, to the filtrate line, to the wash water line and to the compressed air line.

Cake formation occurs during the period of submergence of a section of the drum in the slurry tank. As this section emerges, additional filtrate is sucked from the cake until the sector comes under the water spray. At this time, the connection for the sector changes, through the automatic valve, from filtrate receiver to wash water receiver. When the sector has passed the water spray, it remains under vacuum for a short time

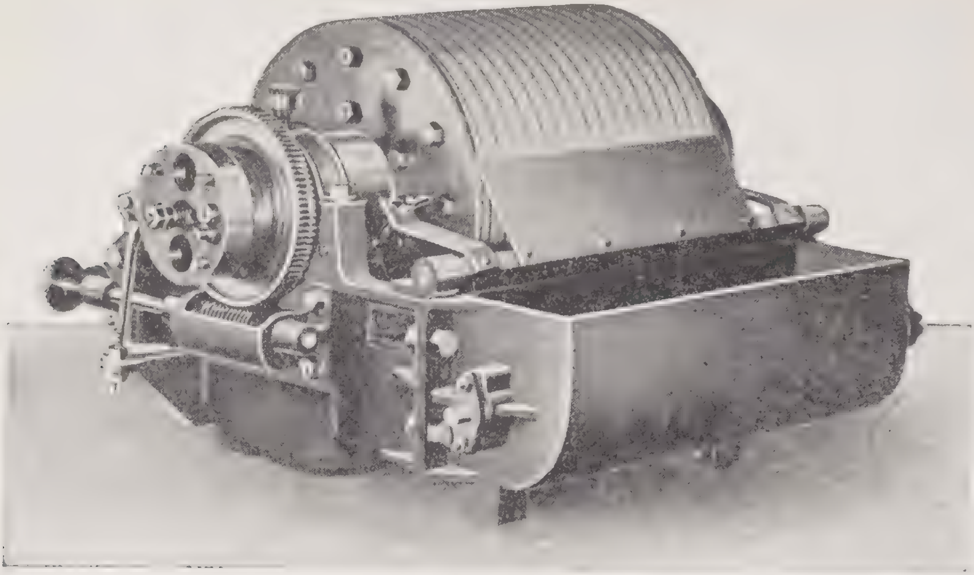
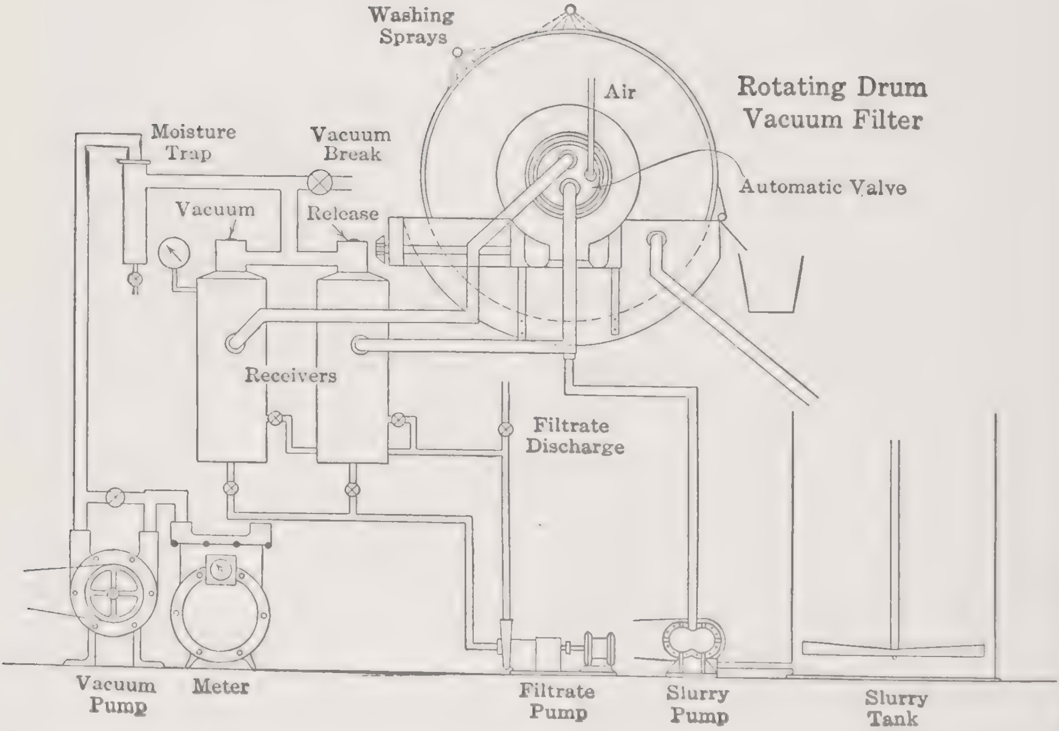


FIG. 3.



Washing Sprays
Air
Rotating Drum Vacuum Filter
Moisture Trap
Vacuum Break
Automatic Valve
Vacuum
Release
Receivers
Filtrate Discharge
Vacuum Pump
Meter
Filtrate Pump
Slurry Pump
Slurry Tank

FIG. 4.

and the cake is dried. Air pressure is then applied so that the cake is loosened from the filter media and can be easily removed by the scraper.

The sectors are covered with a layer of filter cloth which has been wound around the drum and is held in place by a wire, wound spirally around the drum with the windings about one-half inch apart. The wire also prevents the scraper from coming in contact with the cloth.

The vacuum pump connects at the top of the receivers for filtrate and for wash water. The vacuum release valve is operated by a float which prevents flooding of the pump at any time. The by-pass across the pump is used to control the vacuum. The vacuum break is a cock valve and allows the test to be stopped instantaneously so that samples of the cake may be taken at several points in the washing and dewatering stages.

Each receiver tank has a gauge glass so that the quantity of filtrate and the quantity of wash water may be measured at any time.

Procedure. Start the slurry pump. As soon as the tank containing the filter drum is filled so that slurry flows back to the slurry tank, start the filter drum rotating, turn on the vacuum pump and begin the collection of filtrate and wash water. Record the quantity of filtrate collected per unit of time. Measure and record the cake thickness at various points on the circumference of the drum. Measure and record the portion of the drum circumference submerged at any one time. This is the instantaneous filtering area.

Collect samples of the filter cake the entire width of the drum, (1) just above the surface of the slurry (2) midway between this point and the water spray and (3) just prior to the water spray. Analyze these for moisture content and water soluble solids.

Collect three samples, at similar locations after the water spray and make the same determinations.

Determine moisture content of the cake as removed by the scraper.

Repeat the test at two other speeds, increasing the speed of rotation of the drum by fifty per cent each time. Take the same data as in the first test.

REPORT

Include in the record of the experiment:

a. description of the slurry
b. vacuum, inches of mercury
c. room temperature
d. barometric pressure
e. area of drum surface
f. fraction of drum surface submerged in slurry
g. fraction of surface in air drying stage

and for each run

- h. revolutions per minute of the drum
- i. tabulation of time, weight of filtrate, and volume of air from vacuum pump
- j. total weight of cake discharged
- k. specific gravity of filtrate and slurry
- l. gross, tare, and net weights of samples for moisture determination before and after drying.

Compute, from the results of these tests, a satisfactory operating schedule for the filtration of 5000 gallons of a similar sludge in five hours. State the filter area required, with width and diameter of filter drum.

From the per cent moisture in the slurry, filtrate, and cake as it emerges from the slurry, and from the specific gravities of slurry and filtrate, calculate v, the volume of cake per pound of filtrate, for each run. Make a graph of $P\psi/(W_rN/A)$ as ordinate versus (W_r/A) as abscissa, utilizing the filtration data from the three runs. Determine the value of r and b.

From the per cent moisture in each of the cake samples taken from the air drying surface of the drum, and the ratio of soluble solids to water in the filtrate, calculate the weight of filtrate per unit weight of insoluble solids in each sample of cake. Plot this ratio against the distance around the drum from the slurry level for each run.

Tabulate the r.p.m. of the drum, weight of air drawn through the cake per pound of filtrate and the theoretical isentropic horsepower required per pound of insoluble solids in the cake.

From the rate of drum rotation and the fraction of the area submerged in the slurry, calculate the time each point on the surface was in the filtering zone. With this value of θ, the value of b found above, and the values of r, s, and v obtained from leaf tests, calculate the weight of filtrate that should be obtained per revolution of the drum. Compare with that actually collected.

What fraction of the overall resistance is composed of cloth, undischarged cake, and filtrate leads combined? How would you expect this to compare with the similar fraction for a commercial size installation?

Discuss the applications of continuous filters and give their advantages and limitations.

Referring to the curve of weight per cent water in the air dried cake versus w_a/W and the value of k determined in vacuum leaf tests, estimate the ratio of ψ_a/ψ required to give the weight per cent of filtrate found in the cake discharged in each run. Compare this value with the ratio of the experimental filter.

Suggestions for Other Experiments on Filtration

The variables studied may, of course, be investigated on types of filtration equipment other than those specified in the experiments described. In addition to rearranging the variables to be studied and the equipment used for the experiments, there are many other interesting studies, some of which will be mentioned.

The amount of dissolved solute in a slurry often has a marked effect on the cake resistance. A 4% slurry of filter-cel in distilled water to portions of which are added weighed amounts of NaCl to give solutions varying from 0.0001 to 1.0 N provides a specific case.

The effect of other variables on the slurry such as time of aging, method of agitation and method of precipitation may be studied by determining the resistance of the cakes deposited by filtration.

As explained on page 352, washing on a continuous rotary vacuum filter follows a mechanism different from that used with a submerged leaf. The investigation of the washing efficiency of a rotary filter based on that of a submerged leaf would provide useful engineering data. Of interest in the design and operation of a rotary filter are the effect of the vacuum on the capacity and power requirements, the ratios of washing and air drying surface to submerged surface, the method of applying the wash water, etc.

For slurries giving relatively compressible cakes, it is, in general, good practice to increase the filtration pressure gradually. Plugging of the cloth is thus minimized and a more porous cake results. The pressure may be increased to maintain a constant rate of filtration or by means of control devices in any manner which proves the most advantageous. The theoretical treatment of these cycles is less satisfactory than for the constant pressure cycle, but there is little doubt of their practical importance.

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CHAPTER X

CLASSIFICATION AND CONCENTRATION OF SOLIDS

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REFERENCES

Badger and McCabe, Elements of Chemical Engineering.
Walker, Lewis, and McAdams, and Gilliland, Principles of Chemical Engineering.
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SETTLING OF SUSPENSIONS

When it is desired to separate suspended solids from liquids, processes are resorted to which are often called "Sedimentation" or "Thickening." These are especially desirable procedures when large quantities of materials are concerned—quantities far too great to consider straight filtration. The settling of suspensions is a purely gravitational phenomena and Stokes formula, which expresses the movement of small falling bodies, is applicable. In addition sedimentation processes are ordinarily carried out under conditions of viscous resistance. At the beginning of the settling of a suspension the rate of settling is nearly constant or until the condition is reached when the falling particles begin to interfere with each other. This is manifested in an increase in viscosity and in density of the suspension through which the uppermost particles are falling. These two items and data for the rates of settling will be determined in this experiment.

EXPERIMENT NO. 57

SETTLING RATES

Equipment. Five or six 30-millimeter tubes mounted on a board. One 30-millimeter tube with appropriate side-arm attached. Stormer viscometer. Finely divided solids; quartz, barium sulphate, calcium carbonate. Stop watch. Scales and weights.

Procedure. The general principles of procedure for this experiment should follow along the lines recommended by "Ward and Kammermeyer," reported in the group of papers for the Symposium on Sedimentation, published in *Industrial and Engineering Chemistry*, 1940. The particle size and particle distribution of the solids used, is determined by use of the Kelly tube. The settling rates of five or six concentrations of the solid chosen is determined in the mounted tubes by means of observing the layer of settled particles from time to time. Viscosities of the same materials are determined by means of the Stormer viscometer.

REPORT

Tabulate results.

Follow the principles discussed in the reference mentioned above for making up a final report.

Make a statement of conclusions.

HYDRAULIC CLASSIFICATION

REFERENCES

Lewis, Walker, and McAdams, and Gilliland, Principles of Chemical Engineering.
Badger and McCabe, Elements of Chemical Engineering.
Richards and Locke, Textbook of Ore Dressing.

Classification of materials usually pertains to the separation of solid particles on a basis of size of particles or gravity of the particles. In engineering applications where large quantities of materials are to be classified, hydraulic methods are most important because the use of screens for this purpose is both uneconomical and unfeasible. In hydraulic classification with free or hindered settling principles are encountered and various types of equipment which operate on one or the other of these principles are on the market. Many of these types of equipment may be located in the references given above. Laboratory size equipment for work of this nature is available at a number of manufacturers, who have utilized a range of mechanical devices in their products. They are found mentioned in the literature and trade catalogues. Most of this equipment can also be made in the ordinary chemical laboratory which has machine shop facilities.

EXPERIMENT NO. 58

A Study of Various Hydraulic Classifiers

Object. To determine the respective advantages and disadvantages of the following hydraulic classifiers: Jig, Wilfley table, Dorr and cone classifiers, by studying their operation under varying conditions.

Equipment. Set of Tyler standard screens, crushed quartz and pure litharge, each to pass a 20 mesh screen.

NOTE: The four classifiers discussed here are well sketched on pp. 581-585 in Badger & McCabe *Elements of Chemical Engineering*, Second Edition. On account of the wide latitude in choice of materials and sizes, and variety of auxiliary equipment in different laboratories, it seems not justifiable to attempt detailed direction for construction and assembling of these pieces of apparatus. In addition to these classifiers others such as the Akins or Hardinge could be designed and constructed for this experiment.

a. Dorr Classifier: This is the inclined bottom type of classifier equipped with a rake mechanism for removing the solids which fall to the bottom.

b. Double Cone Classifier: Concentric cones arranged so that the velocity of a rising stream of water may be regulated to meet falling solids.

c. Wilfley Table: Flat table with parallel, longitudinal cleats ending in a horizontal line the whole table inclined at about 3° away from the feed. It is given a longitudinal reciprocating motion.

d. Jig: A V-shaped trough which carries a screen and a plunger within a cylinder so arranged so that a pulsating effect is applied to the suspension.

Procedure. a. The Dorr Classifier: Using a feed of 1:1 quartz and litharge affect a separation. Repeat, first varying the rate of feed, then the speed of the rakes. Make screen analysis of both overflow and coarse material in each case. Determine the ratio of the quartz to litharge by dissolving the litharge in an alkali and weighing the residue.

b. The Cone Classifier: Repeat the above procedure letting the variables be rate of feed and rate of addition of hydraulic water.

c. Wilfley Table: Set the table in operation adding a feed of 1:1 quartz and litharge as before. Collect 4 samples and analyze for size and gravity separations. Repeat with a different rate of feed.

d. Jig: (Batch operation).

Place about three inches of 1:1 quartz-litharge mixture on the screen, fill the apparatus with water, allowing a small continuous overflow and start the eccentric which gives the water a pulsating effect. Make a screen and gravity analysis of the overflow and the dense fines. Separate the residue into two layers; screen them and make a gravity analysis on the various screenings. Repeat using a different rate of addition of hydraulic water.

REPORT

Tabulate your results. Which of the above is the simplest type? In which of these classifiers do the laws of hindered settling apply? Discuss the laws of hindered settling as applied to these classifiers. On the basis of these laws explain the result of your analyses for each of the four classifiers, particularly with regard to the residue left on the screen in part (d).

Compare the four types as to adaptability for size separation or density separation, and justify your answers.

In each case discuss the effect of the variables introduced on the efficiency of operation.

LEACHING

REFERENCES

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The process of leaching as studied here is that phase of extraction principles which deals with the removal of a soluble constituent from a solid by means of a liquid solvent. This distinguishes it from a typical all-liquid process. After the removal of a soluble substance by means of a suitable solvent it is generally necessary to recover or separate, either by evaporation or other means, the components of the new system, i.e. the solvent and the soluble material which was extracted. Although that is a separate problem, it is important because it is generally necessary to predict the most economical scheme for the leaching process from the standpoint of the succeeding operation.

In batch leaching or extracting there is an alternative of using one quantity or successive quantities of fresh solvent to attain a given amount of extraction. If it is necessary to prepare a stronger solution, countercurrent processes are adopted. In certain instances a single leach is used and has a very definite value.

In applying mathematical calculations to the process of leaching it is necessary to make certain generalized assumptions as to the condition of operation. For instance the amount of solvent that is carried away by the extracted solid mass must be considered, also the amount of soluble solid that is not removed is important. The question of equilibrium conditions in a system arises and must be considered.

In part I of this pair of exercises on leaching, a purely hypothetical condition is considered—a synthetic mixture of sand and sodium chloride is made up and treated as a substance to be leached. In the second part of the experiment a more practical, yet more difficult scheme of leaching zinc ore with dilute sulphuric acid is considered.

The value of the first procedures lies in its simplicity of operation and the opportunity afforded the student to study strictly the mechanism of the extraction process without being concerned with side issues of lesser importance. The value of the second part lies, in addition to a study of extraction principles, mainly in the opportunity given the student to apply the results obtained to a preliminary pilot plant investigation. Other typical procedures could be adopted for this program, such as those indicated in Badger and McCabe and elsewhere in the Chemical Engineering literature. The causticizing reaction is suggested as a possibility which will give students an opportunity to use initiative in devising a laboratory experiment.

EXPERIMENT NO. 59

Leaching a Sand-Sodium Chloride Mixture

Object. To study the principles governing leaching by comparing the results obtained by leaching a mixture of sodium chloride and sand.

Equipment. Clean sand, sodium chloride (Packer's salt), 3-5 liter tubs having outlet valves about 3 inches from the bottom, a stand on which to place the tubs so that the outlet of the first tub can drain into the top of the second tub and the outlet of the second into the third.

Procedure. A. Successive treatment with fresh solvent (using one cell only): Prepare a 10% by weight mixture of sodium chloride in sand. Place a weighed amount in one of the tubs so that the top level of the mixture is below the outlet. Now measure the amount of liquid necessary to fill the tub just to the bottom of the tap (this amount of water will be left in the tub each extraction so determine it quite accurately and use the same figure throughout). Now add to this a volume of water equal to that just added, agitate well, and allow to settle. Draw off clear liquor and titrate the sodium chloride with standard silver nitrate (for method see any quantitative chemistry text) to determine the per cent of the total sodium chloride removed. Repeat the above, adding first 2 then 4 times as much water, taking a fresh batch of sand and sodium chloride each time. Thus far you have carried out the operations with one treatment and solvent ratios (volume removed/volume retained) of 1, 2, and 4.

Repeat the above three steps but add the water in two equal portions instead of all at once, that is, wash each batch twice with equal quantities of water. Determine sodium chloride in both extractions in each case. (NOTE: Keep in mind that some of the steps in the two procedures here are overlapping.)

B. Multiple, continuous, countercurrent extraction:

Arrange the three tubs on the stand so that the clear liquid from the first tub will flow into the second, etc. Place sodium chloride-sand mixtures in tubs as before and fill each to the valve with water. Now add an equal volume of water to the first and second tubs, agitate, settle, draw off the clear liquid, and discard. Repeat again with the first tub. The above steps have placed the system in a ratio of concentrations such as would exist if the system were continuous, that is, each quantity of the mixture being washed 3 times in countercurrent flow.

(a) Now proceed by adding to the first tub water equal to that already there, agitate, settle, and draw off the clear liquid into the second tub, keeping out a sample for analysis.

Agitate, settle, and draw liquor from the second tub off into the third tub, analyzing a sample.

Agitate, settle, and draw liquor from the last tub and analyze.

(b) Using the same system (same sand and sodium chloride mixtures) repeat part (a) using fresh water. This will give the effect of a 2 treatment, 2 solvent ratio system when combined with the data of part (a) which is a one treatment, one solvent ratio system.

REPORT

1. Fill in the tables.
2. Compare the efficiency of the 1, 2, and 4 treatment runs in the batch system.
3. Compare the efficiency of B (a) and the one treatment, one solvent ratio batch operation of Part A.
4. Compare B (b) and the 2 treatment, 2 solvent ratio batch operation of Part A as to efficiency of leaching.
5. Develop an equation for the calculation of the concentration of solute in each tub of a continuous countercurrent leaching system (assume solvent ratio constant in all cells). See references No. 1 and 2. From this equation calculate the concentrations in B (a) and compare with the values determined.
6. Give several commercial applications of leaching processes.

TABLE 1

No. of Treatments	1		2	
Solvent Ratios	Vol. of Soln Removed	NaCl Extracted	Vol. of Soln Removed	NaCl Extracted
1			(a) .... (b)	(a) .... (b) ....
2			(a) .... (b)	(a) .... (b)
4			(a) .... (b)	(a) .... (b)

TABLE 2

Number of Treatments	Solvent Ratio	Vol. of Soln Removed	Conc. of Soln from Cell			Total % NaCl Extracted
			1	2	3	
1	1					
2	2					

EXPERIMENT NO. 60

Leaching a Zinc Ore

Object. To acquaint the student with a commercial process requiring leaching, and a solution of the problems encountered.

Equipment. *Pachuca Tank:* A lead lined tank about 6 inches in diameter and 10 liters capacity, jacketed with an outer heating chamber. The base should taper at 60° to 1¼ inch opening, a spigot at the bottom admitting air for agitation. (See Badger & McCabe, "Elements of Chemical Engineering," Fig. 193.) Zinc calcine; 66° H_2SO_4; analytical reagents.

Procedure. The charge for the Pachuca tank is made up as follows:

1. 6 liters of head acid (125 cc. 66° H_2SO_4 made up to 6 liters with H_2O).
2. 5 grams $Fe_2(SO_4)_3$.
3. 10 grams of MnO_2.

The air is then turned on to give violent agitation. Steam is turned on in the heating compartment in order to maintain a temperature of 45° C. Zinc calcine is then added to the agitated acid, slowly to prevent lumping and clogging at the bottom. Enough calcine must be added to neutralize all acid, precipitate all iron in solution as $Fe(OH)_3$ and give good settling and coagulation. This may mean from 300 to 700 grams of calcine, the zinc content of which has been determined analytically. The iron content in the head acid and the zinc content in the neutral leach pulp are the important factors in removing impurities, settling, and filtration, and may have to be varied in each case to determine the most favorable conditions. Normal conditions call for about 1.0 g./liter Fe and 25 to 30% Zn.

For a high grade calcine with about 60% Zn, the leach pulp is generally kept at about 30%, while for a low grade calcine (40% Zn), the leach pulp may be kept at 20% Zn.

The extraction in the neutral leach generally varies between 55 and 65% of the Zn in the calcine.

Most leaches have a tendency to foam badly at the start. This can be kept down by the use of a small stream or jet of water or steam. The leach may take from 10 minutes to an hour and a half. The end is indicated by a lessening in the foaming, a darker colored foam, and a different sound in the running of the Pachuca. Samples are removed from the leach with a 100 cc. pipette which has the delivery tube cut off close to the bulb. A part of this sample is filtered (10 cc.), a small amount of HNO_3 (a drop) added to the filtrate (to oxidize any iron present) and 3 or 4 drops of KCNS solution. A deep red color indicates the presence of iron, showing that the leach is not done. This color should be distinguished from that due to an excess of HNO_3, or from color due to copper in solution. The sludge from the filtered sample is assayed for zinc.

NOTE: In the usual practice of zinc leaching this dilute acid leach is followed by a strong acid leach. Since this is a unit operation experiment there is ample opportunity in the experiment described here to afford a good study of the leaching process.

Analyze the filtrate for zinc content by titrating hot with standard $K_3Fe(CN)_6$ solution using 1% ammonium molybdate as an indicator.

Analyze a sample of the original calcine for zinc as follows: treat 5 gms. of the calcine with 5 cc. conc. HNO_3, dilute to 25 cc. and heat for one minute. Add NH_4OH in excess to precipitate iron (most of the other impurities will also precipitate with the iron) and filter. Re-dissolve $Fe(OH)_3$ in HNO_3 and reprecipitate and filter (this precipitation is to prevent retention of zinc by occlusion). Make the combined filtrates slightly acid and pass in H_2S to complete precipitation of copper. Filter and titrate hot with standard $K_4Fe(CN)_6$ as above.

To carry the experiment to a logical conclusion in order to prepare the material for electrolysis proceed as follows: The material in the Pachuca is removed, settled and filtered. The neutral solution is agitated mechanically with zinc dust for the removal of Cu and Cd. The amount of zinc added is generally calculated from the Cu content, which can be determined in a short time by KI and standard thiosulphate. The Cd is usually ignored in this calculation. In most cases, 2 to 2.5 g liter of zinc dust are used. The temperature of the purification *must* be kept near 45° C. Higher than 50° C. will prevent the Cd from precipitating, or will cause it to go back into solution. Half an hour after the addition of the zinc dust, 25 cc. of the solution may be removed, filtered, and tested for Cu and Cd by the addition of 2-3 drops of concentrated HCl and 2-3 drops of Na_2S solution. A clear or milky white solution after standing a minute shows the purification to be done; a yellow or brown orange shows that all Cu or Cd have not been removed. The time ranges from one to two hours, depending on the speed of agitation and the amount of excess zinc dust. When done the precipitate is filtered and the solution is ready for electrolysis. (Na_2S solution is made up by dissolving 1 lb. Na_2S in 2 liters of H_2O. 40 cc. of this strong solution is diluted to 300 cc.)

REPORT

Write up the experiment giving a detailed description of your procedure.

Determine the per cent of zinc recovery if the electrolysis recovery were 100%.

Using the references listed above give the reactions which occur

1. In the leaching.
2. When the zinc dust is added.

What purpose does the MnO_2 serve?

Why can complete removal of the iron be taken as an index of complete reaction?

Give a brief outline or flow sheet for the production of zinc by this method, from the time the ore leaves the mine until the pure metal is obtained.

FLOTATION

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Like some other industrial processes, flotation has hitherto been developed almost entirely on an empirical basis, with relatively little practical contribution from the fundamental chemistry and physics, on which the phenomena are based. In fact, its development by the trial-and-error method has been perfected to such a high state that it is becoming generally thought that further important advances can be brought about only by the application of a knowledge of the mechanism of the process. Naturally, such a complicated process has brought forth numerous theories, most of which have failed to withstand the test of time. It is significant that no theory has yet been advanced which is universally accepted.

The theories, which have been given much thought, are the following: "electrical" theory, "contact-angle," "gas" theory, "adsorption" theory, and the "chemical" theory. The last two appear to be the best to explain the phenomena of "flotation."

This experiment is divided into two parts; one deals with the separation of a relatively simple mixture of minerals, while the other is more complex. In part one, by the addition of certain specified chemicals, classed as collectors, adjusters, and frothers, it is possible by flotation to improve the desirable mineral content of a given ore such as low lime rock.

In part two there is a system containing a number of minerals to which the principles of flotation may be successfully applied in order to separate the minerals from one another. In addition to the reagents used in the first part, compounds classed as depressors are used. This results in a successive means of separating groups of one or more minerals which may be found in nature. A rather difficult laboratory procedure for the analysis for different minerals is necessary here. It is desired to make a quantitative study of flotation methods.

EXPERIMENT NO. 61

Flotation of Calcium Carbonate

Object. To determine the conditions most suitable for the optimum concentration of $CaCO_3$ in a low lime rock by a study of the variables entering into the process and to study the general principles of flotation as applied in the concentration of lime rock used in the cement industry.

Equipment. Cresylic acid, oleic acid, low-lime rock (containing 30-40% $CaCO_3$), motor (variable speed), compressed air, air agitation flotation machine as illustrated in Fig. 1.

Container is rectangular and may be made of wood or sheet metal. The size is optional, one of 2-5 liters capacity will give quite satisfactory results providing the agitator is strong enough to give the contents sufficient motion to keep the solids in suspension. The propellers for removal of the foam may also be run by the motor.

Procedure. Grind the limestone to pass a 200 mesh sieve. Analyze a sample for $CaCO_3$ by the method given below. Fill the cell about $\frac{2}{3}$ full of water. Add enough of the powdered limestone to form a 15% suspension by weight, and start the agitator, running it fast enough to keep the solids in suspension. Now add about 0.5 gm. oleic acid per 100 gm. lime rock while continuing the agitation. This acid acts as a collecting agent. Now add a quantity of cresylic acid (the frother) equal to the amount of oleic acid added and turn on the air sufficiently to produce a good foam. Turn on a slow stream of water to replace that leaving as foam. Continue the agitation until the foam appears to carry no more solids. Filter a portion of the concentrate, dry and analyze for $CaCO_3$. Repeat the above on the residue left in the tank.

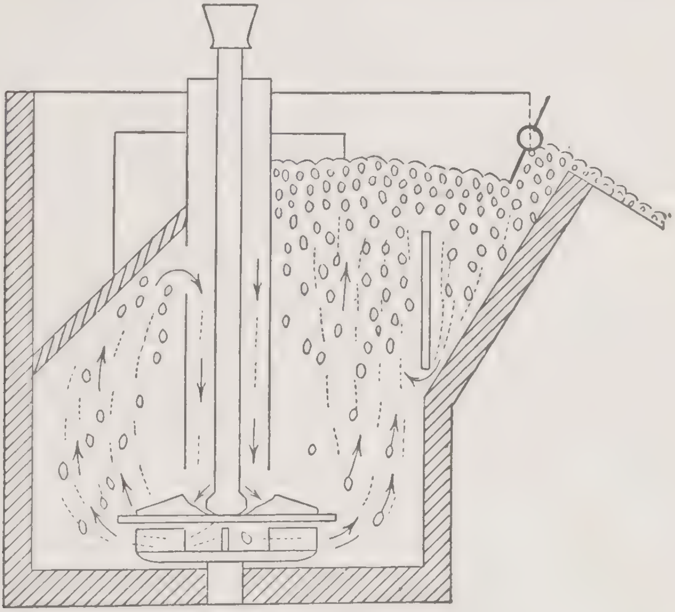

FIG. 1.

Repeat the first part using first 0.2 gm. oleic acid and 0.7 gm. cresylic acid per 100 gm. feed, then using 0.7 gm. oleic acid and 0.2 gm. cresylic acid per 100 gm. feed.

Sketch of Cell. Shows method of aeration, circulation, and removal of concentrates. All air is drawn down from the standpipe by the rotating impeller. As impeller rotates below the stationary hood the pulp, reagents, and air are mixed, thus producing a selective froth. The paddle-shaft then removes the froth (concentrate) at once which is important in recovering mineral. There are no cross or interfering currents.

APPROXIMATE METHOD FOR ANALYSIS OF $CaCO_3$

Place a weighed quantity of the dried material to be analyzed in a beaker. Add a *measured* quantity of standard (0.1 N) HCl (sufficient to react with all the carbonate present). Boil for two minutes to insure complete reaction. Titrate the excess HCl with standard (0.1 N) NaOH and by loss of HCl calculate the per cent of $CaCO_3$.

REPORT

Tabulate your results.

Would it be advisable to have two cells in series? Why?

In what proportions should the collecting agent and the frother be used according to your results? Explain.

Discuss the theory of flotation.

EXPERIMENT NO. 62

Differential Flotation

Object. By regulation of the variables (reagents, time, stirring, etc.) to determine the set of conditions necessary for the best separation of two ores ($CuFeS_2$ and ZnS) by differential flotation.

Equipment. Gangue, sphalerite (ZnS) and chalcopyrite ($CuFeS_2$) to pass a 150 mesh screen, sodium cyanide, sodium carbonate, cresylic acid, thiocarbanilid, pine oil, copper sulfate, potassium amyl xanthate, air agitation flotation machine (described above).

Procedure. Suggested proportions of chemicals per 100 gm. ore charged: 0.25 gm. sodium carbonate, 0.01 gm. sodium cyanide, 0.02 gm. thiocarbanilid, 0.005 gm. cresylic acid, 0.005 gm. pine oil, 0.1 gm. $CuSO_4$, 0.02 gm. potassium amyl xanthate, and 0.005 gm. pine oil.

The sodium carbonate is added to give correct alkalinity, the sodium cyanide deactivates the sphalerite by forming a coating which is wetted by water, cresylic acid and pine oil are frothing agents, the thiocarbanilid increases the selectivity of copper from zinc. After the chalcopyrite is removed the $CuSO_4$ is added to destroy the effect of the depressing agents, the pine oil is a frothing agent, and potassium amyl xanthate is a collecting agent.

Fill the cell two-thirds full of water and add a mixture of gangue, sphalerite, and chalcopyrite in the proportions 5: 1: 1, making about a 15% suspension by weight. Also add the sodium cyanide and sodium carbonate: agitate. Add cresylic acid and thiocarbanilid and turn on the air while continuing agitation. Allow enough fresh water to enter to replace that lost. Run until the froth carries no more solids. The froth carries the copper concentrates.

Shut off the air, add the copper sulfate and potassium amyl xanthate while continuing agitation. Add the pine oil and turn on the air. The froth will carry the zinc concentrates.

By varying the amounts of the chemicals added and the conditions of operation, the following problems can be solved:

1. Correct amount of sodium carbonate to give right alkalinity.
2. Proper amount of sodium cyanide.
3. Sufficient thiocarbanilid for highest degree of selectivity.
4. Just enough cresylic acid, pine oil, and xanthate to give a good froth and collection of sulfides.
5. Removal of practically all of the chalcopyrite in the copper circuit before adding reagents for the zinc circuit.
6. Sufficient stirring to keep the ore suspended.
7. Sufficient time for reagents to react with the pulp before a concentrate is collected.

Analysis of the Ores and Concentrates

Treat 0.5 gm. of the ore or concentrates with 5 cc. of concentrated nitric acid in small evaporating dish covered with watch glass until vigorous action is complete. (It may be necessary to place on heated sand bath to start reaction.) Dilute with water until dish is half full. Heat nearly to boiling with stirring to get copper and zinc salts into solution. Wash into 150 cc. beaker (volume 50-70 cc.). Add ammonium hydroxide in excess to precipitate ferric hydroxide. Heat to boiling. Filter on Hirsch funnel with suction. Redissolve precipitate of ferric hydroxide with the filter paper in small amount nitric acid and water. Reprecipitate the ferric hydroxide. Filter. Add filtrate to preceding one. Discard the precipitate. (The special solution and precipitation is to prevent the ferric hydroxide from retaining a large amount of zinc and copper ions by occlusion.)

Zinc and copper exist in the filtrates as complex ammonium ions. Make the combined filtrate slightly acid with nitric acid as marked by the solution becoming practically colorless. Heat to boiling. Pass in H_2S to precipitate copper. Filter. Test the filtrate to make sure all copper is precipitated. The combined copper precipitates are dissolved in concentrated nitric acid as was the original. The filtrate from the H_2S precipitation, containing the zinc, is caught in a 600 cc. beaker and is titrated while hot with standard potassium ferrocyanide solution, using a 1% ammonium molybdate solution as an indicator, to obtain the percentage of zinc.

The redissolved copper sulfides are diluted to 50 cc. and filtered to get rid of sulfur and the filter paper. Wash thoroughly the residue to be sure all copper ions are removed. Make just alkaline with ammonium hydroxide. Evaporate down to 30 cc. Add acetic acid in slight excess. Determine the per cent copper volumetrically by adding KI and titrating with standard $Na_2S_2O_3$ solution.

REPORT

Tabulate the results.

Discuss solution of the seven problems listed, as determined from your data.

ELECTRICAL PRECIPITATION SOLIDS FROM GASES

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Cottrell, in America, has been the leader in developing the commercial application of the principle of electrical precipitation from gas streams of solids or liquid drops in suspension. In the operation of this principle a unidirectional, high potential electrical current is maintained over two electrodes which are insulated from each other. The electrodes are placed so that the gas to be treated can pass between them. In passing through a highly charged electrical field the particles in suspension are ionized and precipitated on one of the electrodes, from which the precipitated solid can be readily removed. Although this experiment is essentially qualitative in character, a quantitative character can be introduced by modifying the method given below to include measuring air used and solids precipitated.

EXPERIMENT NO. 63

Object. The purpose of this experiment is to give the student an opportunity to set up a simple Cottrell apparatus and see how and why it functions. He will also make a study of the variables entering the process by making several trial runs.

Equipment. Wash bottles, rubber and glass tubing connections, copper wire, iron pipe 1 in. × 18 in., 2 mm. brass rod, ring stand, clamps, glass tube 1 in. × 18 in., burner, two 6 volt batteries, conc. HCl, conc. NH_4OH, conc. H_2SO_4, voltmeter, ammeter, rheostat, spark coil. See Fig. 2.

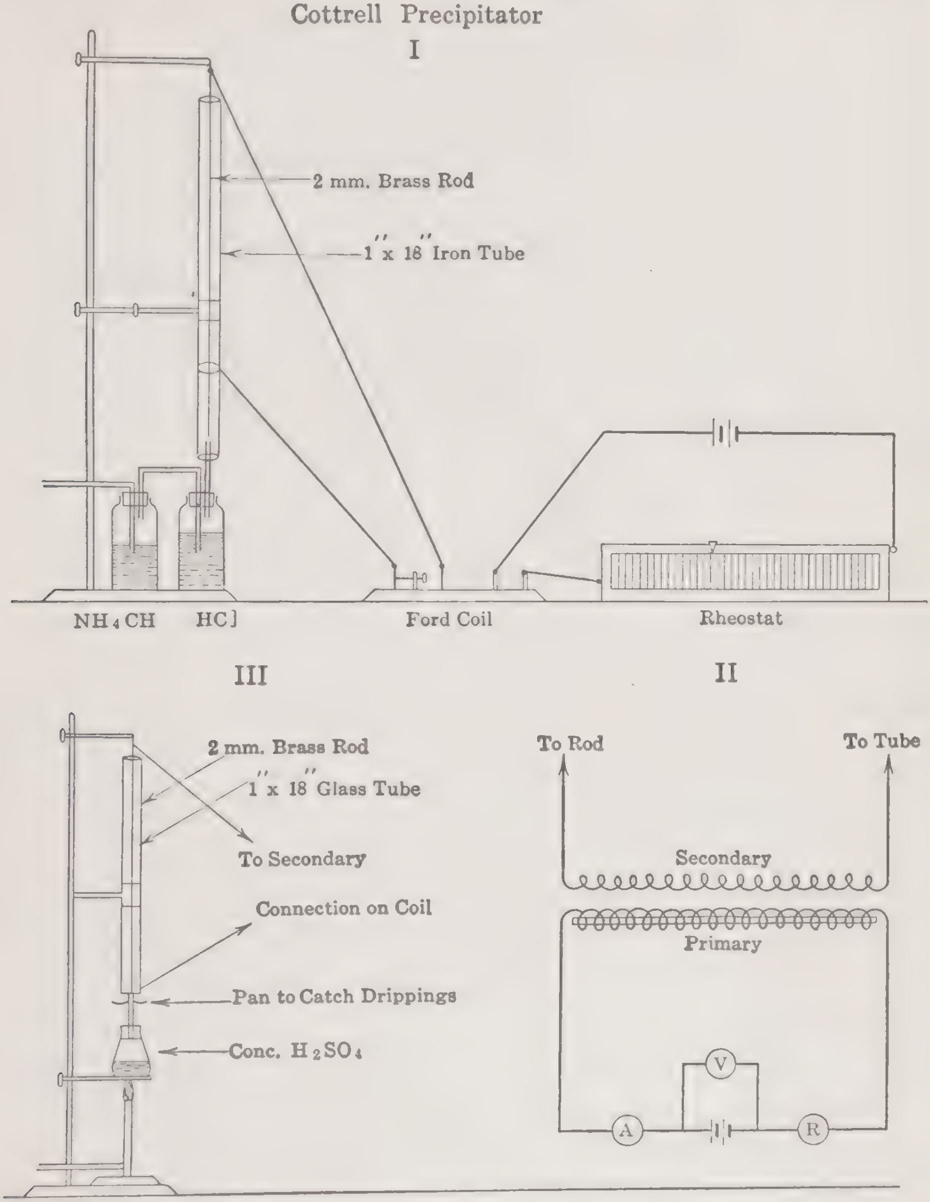

FIG. 2.

Procedure. The apparatus in diagram I is set up as follows: The 1 in. × 18 in. iron pipe is connected to one terminal of the secondary of the spark coil. The other terminal of the secondary leads to a brass rod about 2 mm. in diameter extending down through the middle of the pipe (care must be taken to place the rod in the exact center of the pipe in order to get a uniform discharge). The end of the rod should be fused to a small ball to eliminate the point which would favor the formation of a spark at this point. Two six volt batteries supply the current and are connected in series with an adjustable resistance to the primary of the spark coil.

Diagram II shows the scheme for connecting the spark coil to batteries and precipitator. If the two six volt batteries are hooked up in series they will give twelve volts in the primary of the coil when the resistance is a minimum. This voltage will give an e.m.f. of 12,000 to 16,000 volts in the secondary (approximately equivalent to a one-half inch spark gap).

When the apparatus is properly assembled, with the minimum resistance of the rheostat and the circuit closed, there should be a spark between the center rod and the wall of the pipe. (The spark gap on the coil should be adjusted as fine as possible in order to cause as even a flow of electrons as possible. Care must be taken, however, not to get the adjustment so fine that overheating of the points occurs.) The resistance in the rheostat should then be increased until the sparking is just stopped while the coil point still continues to vibrate.

The smoke is produced by allowing compressed air to flow first over conc. NH_4OH and then over conc. HCl. If the smoke is not dense enough the air may be allowed to bubble through the two liquids. The white fumes of NH_4Cl, blown into the bottom of the pipe, issue from the top in a cloud. With a steady stream of the chloride entering at the bottom its emergence at the top can be controlled at will by completing and breaking the primary electrical circuit. It is desirable to have the tube in the HCl bottle as large as convenient to prevent clogging. A little experimentation will be needed to adjust the air pressure to the needs of the particular set-up. After the precipitator has been in operation for some time solid NH_4Cl may be shaken from the tube by tapping it. Dry the tube thoroughly before each run so that the finely divided precipitate will not dissolve on contact with the pipe.

Diagram III shows a modified form in which the precipitation is made visible by use of a glass tube. This is quite striking for in the dark the corona may be seen by those close at hand. This glass tube, used in place of the iron pipe, is made a conductor by moistening the inside with conc. H_2SO_4. The wire leading to the tube is extended to the inside wall to make contact with the film of H_2SO_4. Fumes of hot conc. H_2SO_4 (SO_3 fumes) are blown up the tube and can be completely stopped by completing the circuit of the precipitator. A circular metal tray may be made to catch the acid as it drips from the bottom of the glass tube.

Make three runs of five minutes each in each of the above set-ups, recording the voltage, amperage, and weight of product received in each case. Additional valuable data could be obtained by determining the amount of solids in the gas stream, i.e., the concentration of solids.

REPORT

Tabulate the data. How do the three runs agree? If they do not agree, why not? How could the variables be controlled so as to get constant results? Calculate the cost of each precipitation at 3 cents per K.W.H. What other items of operating cost would enter into an industrial set-up of this sort?

In each case where was most of the precipitate formed? In what direction do the electrons flow, from pipe to wire or vice versa? What would happen if sent in the wrong direction? Explain what has occurred. Give a complete theoretical discussion of the electrical precipitation of colloids. Name several industrial applications of the Cottrell precipitator.

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CHAPTER XI

SIZE REDUCTION

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The subject of size reduction is one which may be approached from many angles. A massive material may be reduced by the application of mechanical force. Such operations are known as crushing and pulverization, or, more broadly, grinding. Other materials occur in an apparently massive form but are in reality compact masses of fine particles held together by bonding forces which may be much less than those which hold together the parts of the unit particles. The reduction of such materials, often referred to as grinding, may better be called disintegration. The line of demarcation is, however, not a sharp one and it is in the more extreme cases that one can best illustrate. A lump of quartz is homogeneous and it breaks down in a distinctive way when ground. Mica schist and some of the ore bearing minerals are composed of individual grains of different minerals and they are heterogeneous. Such materials, if well bonded, break into fragments on grinding in a manner simulating that of homogeneous material; but if the binder is relatively weak, the material may disintegrate very largely into the unit particles of which it is composed. A dry filter cake may be the extreme of heterogeneity and the problem of disintegration may be to eliminate the cake form and to secure a powder of the unit particles.

In many cases it is not desired to exert mechanical force upon the material. Alternatives exist, such as conveying the material in a rapidly moving air stream and projecting it sharply onto an anvil, saturating pieces of the material with water under pressure and exploding them with the release of pressure, heating the material to a high temperature and quenching whereby the uneven shrinkage causes rupture, dissolving the material in a suitable solvent or fusion mixture and precipitating under conditions which create a finer size, or dissolving the material under conditions in which it is chemically changed and restoring it to its former composition by chemical reaction, albeit in a different state of fineness.

Such are the examples pertaining to rigid bodies. Two separate fluids, each existing in bulk form, may be intimately dispersed in each other as emulsions. This usually requires some chemical action in the form of an emulsifying agent. The size of the emulsion particles is dependent upon the manner of treatment and upon the physical and chemical properties of the system, including the emulsifying agent.

The laboratory experiments in this section include three phases of this many-sided question of size reduction, namely, mechanical disintegration of solids, emulsification, and precipitation, and they are thenceforth treated separately.

MECHANICAL DISINTEGRATION OF SOLIDS

Devices for the reduction of solids by mechanical means may include:

1. Direct pressure mills.
    - Jaw crusher.
    - Gyratory and cone crushers.
2. Roll crushers and pulverizers.
    - Double and single crushing rolls.
    - Ring, plate, or bowl roller mills.
    - Ball ring or race mills.
3. Revolving cylinder mills.
    - Ball or tube mills.
    - Rod mills.
4. Hammer or beater mills.
5. Disk mills.

Some of these mills, as in the first group, apply direct pressure to cause rupture of the particles. Others, as the hammer mills, operate at high speed to cause crushing by impact. In the production of finer sizes, mills usually operate through a mass of particles, causing particles to break each other under various kinds of interaction. This is necessary for low maintenance cost; for if reduction can be accomplished between particles, wear of the machinery can be lessened and the life of the parts increased.

A number of terms have been used in the literature to express the actions taking place upon the particles during reduction; but for simplicity these may be limited to three: pressure, impact, and attrition. The action of pressure may be likened to the physical testing of a cylinder, except that the faces are not specially prepared to fit the machine. The action of impact differs from it in that the rapidity of striking a blow may localize the stress, and fracture may occur without deformation of the entire piece. Attrition is essentially the action of rubbing and it will, with homogeneous material, tend to wear off corners and leave a smooth residual core, while with soft heterogeneous material, the entire piece may be disintegrated. Mixtures and combinations of these actions occur together in most mills, and they frequently vary in their relative extents with the speed of the mill, the amount of material in it, and the nature of the feed substance.

The character of the fracture differs with different kinds of machines and their condition of operation. The reduction ratio, i.e., the ratio of feed size to size of the largest pieces in the product, has an important bearing. Large reduction ratios tend to produce large quantities of dust, while smaller reduction ratios, taken consecutively, will ordinarily produce much less dust. Frequently the operation of closed circuit grinding is used. This consists of partial reduction of the feed, elimination of sizes over a given diameter by sieving or by settling in a fluid medium, and returning these to the mill for further grinding. For information on sieving the student is referred to Taggart (3) and on settling and inertial separations to Perry (2). The value of closed circuit grinding lies in reduced cost of mill operation and maintenance and in the better control of product size.

Particle diameter has been mentioned and as a term it may be used quite loosely. It may be the aperture of a sieve, or it may be some dimension of the particle observed in the microscope or computed from settling data. In view of this situation, diameter in reference to data should be explicitly qualified. Average diameter is used still more loosely. A ground product consists of many sizes of particles which can be represented as a distribution curve, usually with diameter plotted against some function of frequency, surface, or weight. A mean diameter may be a frequency mean, a surface mean, a weight mean, or even some other type of mean based on statistical considerations. It is fallacious to use a frequency or a weight mean to compare surfaces, and usually resort is had to a process of summation of values obtained over short intervals of diameter in which even the arithmetic mean diameter will suffice. Specific applications of these principles are left for a later part of this section.

The problem of mechanical disintegration of solids resolves itself into three primary phases:

1. Comparative reduction characteristics of materials.
2. Energy requirements and efficiency.
3. Fineness distribution of finished product.

The first of these has been referred to, but it is of interest in an experimental program only insofar as it may be quantitatively evaluated. Hardness may be evaluated in terms of the Mho scale. It has not generally been regarded as a suitable criterion of grinding character or even of mill wear. Recently, Fahrenwald (k) has shown a relation which indicates that more attention should be placed on this. Then, there is the question of size of feed. It is obviously bad practice to place large lumps in a small ball mill or to try to grind fine material further in a device designed to produce a coarser product. These extremes are suggestive of the real problem of inefficiency in a mill. Hence, if a test is used in which some standardized mill action is caused to operate for a definite period of time upon a fixed size and weight of feed, the final product should have some fineness characteristic by which it could be compared with other substances tested under like conditions.

Such a test is that known as grindability; but it has not been a simple test to standardize. It is practically impossible to have completely representative samples of the same size of raw material; and it has been necessary to accept a portion of the material crushed, to a certain narrow size range, as representative of raw material. From the viewpoint of sample, it might be better to use the entire material without separation and measure its size. The test is, however, dependent upon efficient and well-standardized grinding and discrepancies would result if there were a large leeway in feed size. Finally, there is the criterion to be used for comparison and surface has been selected. Two methods have been accepted as tentative standards by the American Society for Testing Materials and either of these methods may be used for evaluating grindability of certain materials. Such tests have been run quite extensively within the past few years. It is suggested that reference be made to data submitted by Hardgrove in a recent paper before the American Institute of Chemical Engineers (8).

The energy requirement for crushing and grinding has been the dominant problem for several decades. While it is associated with size characteristics and grindability, which have not been evaluated until recently, it was long ago studied with a qualitative estimation of these points. The age-old dispute of Kick versus Rittinger (1, 3, 9) has not been fully settled. However, with the tools for quantitative measurement of fineness, Gross and Zimmerley (6) and Martin (7) have shown that new surface produced is the most reasonable criterion for estimating the energy of pulverization.

It then becomes a question as to how efficiently the different mills function to produce new surface and what feeding characteristics will yield the maximum amount of desired surface per dollar expended. It has been found in the ball mill that each unit period of operation gives an equal increase of surface until the fineness becomes such that the effectiveness of grinding is impaired. It has been found that small amounts of moisture affect the point at which a mill becomes ineffective. Small amounts of certain addition agents may extend the region of effective grinding.

There are many ramifications of the energy problem and numerous variables can be studied. Reference may be made to Work (5) for a brief discussion and literature references. Attention should be called to the special point of useful energy and energy to operate the mill. It is not safe to subtract the energy of the mill when operating without charge, from the total energy. For example, in a ball mill, the charge may bring the center of gravity inward toward the axis and actually diminish the driving torque when charged. It is better, when possible, to operate with a maximum amount of energy in the grinding and a minimum amount used by the parts. Practical considerations control the measurement and energy values will have to be restricted to the facilities available with the equipment. Time is sometimes as useful as actual power consumption for making comparisons.

The size distribution of the finished product from a grinding operation is an important factor in its usefulness. In some cases, that property, associated with shape, is tested by the volume of a weighed mass. It is often integrated into a surface value. Sometimes a limited characteristic of this distribution is used, as the percentage passing a No. 200 sieve. The purpose to which a product is to be put determines the nature of the fineness measurements made upon it. If a reaction depends upon a surface function, that measurement alone may suffice. This has proved to be important with both the raw mixture and with finished portland cement Frequently, however, the rate or the completeness of the reaction is determined by the coarsest size of particle. This factor is important in the burning of pulverized coal and in the ceramic industry. Abrasives are usually graded into narrow size ranges, as the maximum size particle determines the character of the finish when the abrasive is used, and the fines are often of little value in that operation. Energy considerations become important as they relate to fineness distribution, since it is a waste of energy to produce finer particles than are actually needed. Oftentimes, various sizes of a ground material are separated for widely different uses; and if more fines are created than can be absorbed in the market for those sizes, the losses may be a serious factor. It is, therefore, essential to develop a concept of grinding and classifying devices to permit a proper choice and proper application. Size distribution curves are also useful as a means of arriving at the laws of grinding.

Depending upon the method of measurement and upon the use to which they are to be put, size distribution curves are represented in any of several ways.

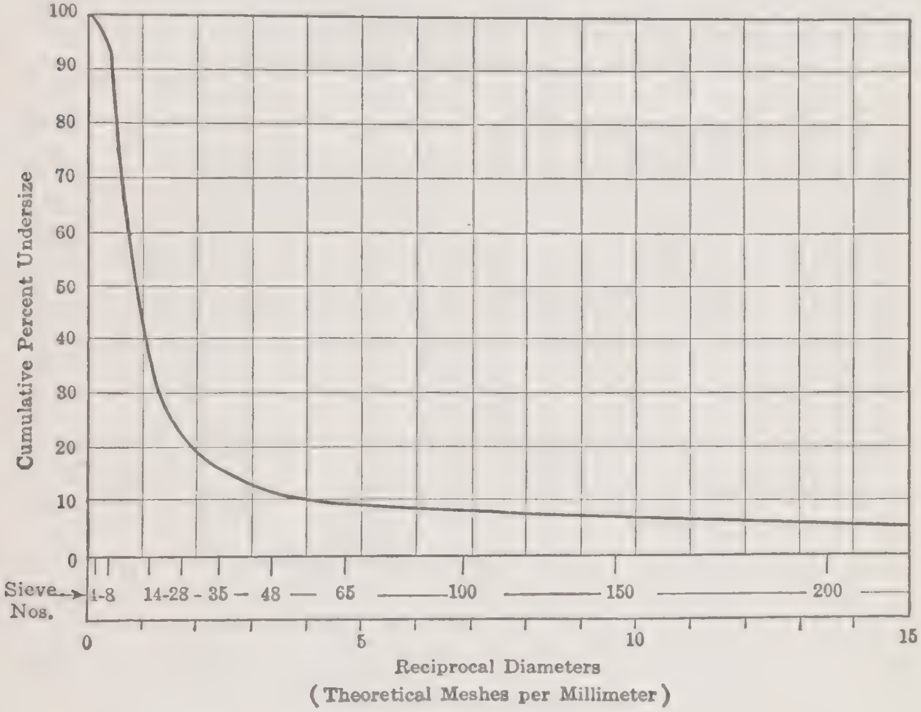

Fig. 1.

Cumulative Plots. It has been the custom to represent sieve data in the mineral industries in terms of cumulative percentage coarser than, or finer than, the stated aperture of each sieve in a series. Such a form is shown in Fig. 1. Regardless of the actual sieves used, the curves for a given material are identical, providing only that the material be properly sieved, and that a sufficient number of sieves be used to define the curve. The plot of cumulative per cent undersize against the reciprocal of diameter constitutes the Gates diagram. It can be shown that the area under this curve is a measure of the surface of a material. This may be used to compare materials and to estimate comparative grinding energies.

Per Cent on Sieve. A more sensitive plot than the cumulative per cent is the per cent on sieve. It is, however, dependent upon the exact sieves used. A standard series such as the American Standard (A.S.A.—Z23.1—1939) which covers the A.S.T.M. and the Tyler series, is most commonly used in a nesting; the apertures of which are increased successively by 2, $\sqrt{2}$, or $\sqrt[4]{2}$. Of these the square root of 2 is most commonly used. It is more sensitive than the 2, and the fourth root of 2 is subject to error unless carefully used. Gaudin (8) has shown some characteristic forms for ground homogeneous and for ground heterogeneous materials when the logarithm of per cent on sieve is plotted against the logarithm of sieve aperture. He has proposed a law for fineness distribution of ground homogeneous materials. Such a plot may be used as a basis for testing the accuracy of individual sieves and for assigning to them a new diameter which will yield a smooth curve. A characteristic form is shown in Fig. 2.

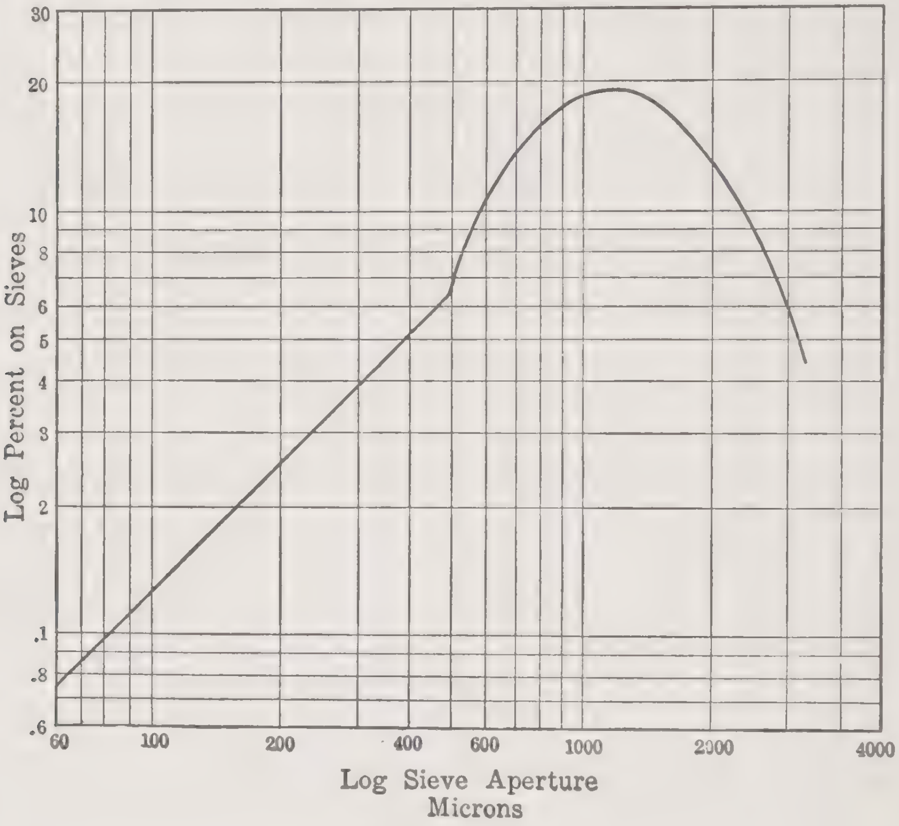

FIG. 2.

Per Cent by Weight per Unit Diameter. The two previously mentioned methods of representation may be used below subsieve size if the proper diametral correlation is effected; but there has been developed for the subsieve range and extended to the sieve range a method of representation known as the per cent by weight per unit diameter. It is commonly expressed as percentage per micron and plotted against the diameter in microns. It may be derived from any source of adequate data by ascertaining the percentage, by weight, occurring between two diameters and dividing by the difference in the two diameters. Thus an average percentage for each and every diameter between these two values is established; and it, with values for other diametral intervals, may be plotted as a histogram or block plot. A smooth curve drawn through these blocks represents the fineness distribution. This curve is independent of diameter, in the same manner as the cumulative plot, and it is sensitive in the same manner as the per cent on sieve. Its accuracy can be verified as the area beneath the curve must total 100% of the weight of sample when uniform coordinates are used. Roller has shown that if this plot be made against the logarithm of diameter the area under the curve is a function of surface. (See Fig. 3.) The proof can be established by simple steps in the calculus.

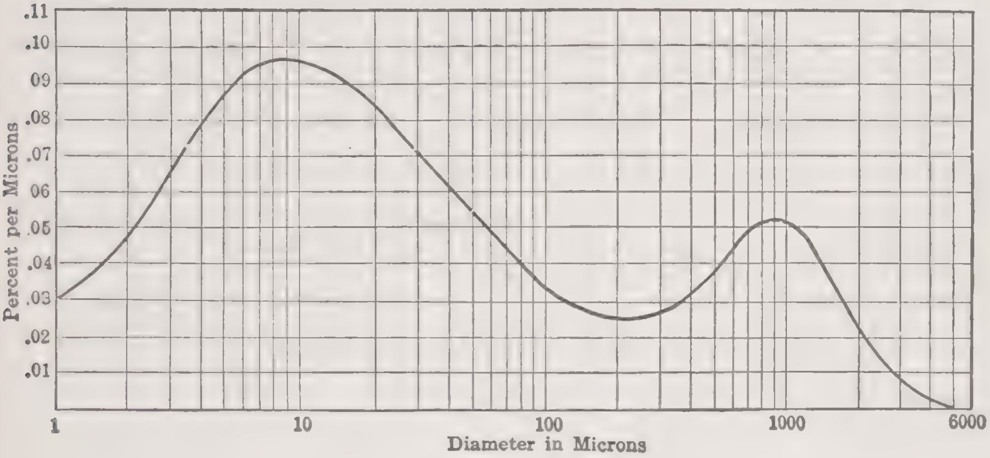

FIG. 3.

This form of data has been modified and presented as per cent frequency and as per cent surface. Work (5) has developed a relation for grinding based on frequency distributions. Other laws of grinding have been developed from statistical forms of distribution curves, but the application of these laws is too complex for general use.

Analytical Determinations. In the determination of grindability of materials, in the comparison of mills, and in the variation of mill operations, the size distribution of feed and product in any run is the criterion of measurement. The method of sampling is important in order that the measurements may be truly representative. In the coarse range of sizes, sieves are used, and the particle diameter is defined in terms of sieve aperture. For the finer materials, elutriation and microscopic measurement are used, and the diameters observed in the microscope may be converted to sieve aperture by a step of calibration. Indications of surface in these fine products may be had by the use of the turbidimeter.

Sampling procedures are necessary because graded products tend to segregate and no one section of the material need be truly representative of the fineness of all of the material. The first requirement in sampling is the size of the sample to be tested. A good rule to follow is that the weight of sample to be tested should be between 100 and 1000 times the weight of the largest particle in the sample, the actual amount to depend upon the accuracy desired. For sampling procedures by quartering, see "Standard Method of Sampling Coal" (A.S.T.M. Standards, 1939, Part III, D21-16, pp. 11-14). For laboratory reduction and sampling, see method (same reference, D271-37, pp. 15-41; also see Perry, pp. 1455-1460). It is well to adopt a definite weight for samples to be sieved, as follows:

Maximum Size	Weight in Grams
1 inch	1000–5000
4760 microns (No. 4 sieve)	200– 500
840 " (No. 20 sieve)	100– 200
149 " (No. 100 sieve)	25– 50

From the mill charge, to this weight of sample for test, the principles of accurate sampling should be observed.

EXPERIMENT NO. 64

Standard Sieve Test

Object. To measure size reduction by standard sieve test.

The analytical procedure of sieving is intended to divide a sample into many fractions closely limited as to size, thereby giving size distribution based on the weights of these fractions.

Equipment. In the first place the sieves used should be of good grade wire cloth and should be free from defects and undamaged. They should conform to the specifications of the American Society for Testing Materials (A.S.T.M. Standards, Part III, E11-39, pp. 535-41 [1939]). Sieves covered by A.S.A. specifications may be used.

Procedure. The procedure involves shaking the sample on a sieve or in a nest of sieves until the amount passing through each sieve in a period of one minute is negligible. As a guide to sieving procedure, reference may be made to the test for powdered coal described in the A.S.T.M. Standards for 1939 (Part III, p. 43, Section 8, through Appendix, p. 45). In some cases, notably ball mill samples, it may be desirable to wash the material through the finest sieve with water, to discard the fines passing through the sieve, to dry the material retained, to recover it and subject it to sieving procedure. When the entire sample is recovered, i.e., when washing is not resorted to, a material balance should show not over one or two per cent loss; and the greater part of this is finer than the No. 200 sieve.

REPORT

Results are expressed as per cent by weight between sieve intervals and per cent finer than the finest sieve used. They may be computed to cumulative values and also to per cent per micron.

To calibrate the microscope in terms of sieve aperture, or vice versa, retain the sample from the No. 200 sieve and, after making the weighing, replace it on the sieve, and tap the sieve over a microscope slide. When sufficient particles have been accumulated on the slide, measure them in the microscope and report the average diameter. Particles of less than one-half to two-thirds the sieve aperture may be ignored as dust; but if they occur frequently, the sieve test has been improperly performed.

If any large percentage occurs less than the No. 200 sieve, sub-sieve methods may be used to measure the distribution of size. Use may be made of the Roller Air Elutriator or the Pearson Air Analyzer or wet elutriation. The hydrometer sedimentation tests (A.S.T.M. Proceedings, Vol. 35, Part I, pp. 953-965 [1935]) and the turbidimeter sedimentation tests (A.S.T.M. Proceedings, Vol. 35, Part I, pp. 777-787 [1935]) are also applicable. The methods involving patented equipment or described in the references may be utilized if the equipment is available.

EXPERIMENT NO. 65

Sedimentation

Object. To measure particle size by beaker sedimentation.

THEORY

Wet separation may be effected with ordinary laboratory equipment or with comparatively inexpensive special equipment. These methods depend upon the falling velocities of particles in a fluid medium; and, for particles below 100 microns (or the No. 200 sieve, 74 microns), the falling rate in liquid media will involve only Stokes' Law. This is expressed in convenient form for use, as follows:

$$V = \frac{D^2(P_s - P_l)g}{18u}$$

where V = velocity of fall, ft./sec.
D = particle diameter, ft.
P_s = density of solid, lb./cu. ft.
P_l = density of liquid, lb./cu. ft.
u = viscosity of fluid, ft.-lb.-sec. units
g = gravitational constant, ft./sec.^{2}

Equipment. The beaker sedimentation test may be performed with 800 cc. or liter beakers. From 10 to 25 grams of material, depending upon its density, is accurately weighed and dispersed with a small amount of water to make a fluid paste. Ammonia, soap, saponin, or water glass may be used in small quantities to insure dispersion of the particles. As the dispersant will depend upon the character of the material, this should be tested by observation under the microscope to see that the particles are not clustered but are free. Observation of a pronounced Brownian movement among 1 or 2 micron particles is an indication of the suitability of the dispersant.

Procedure. The paste is washed into a beaker and water added, to a level near the top of the beaker. The suspension is stirred and when thoroughly mixed is allowed to settle for a predetermined time and then decanted to a definite level, leaving about one-quarter of the volume in the beaker. It is remade, mixed, and decanted successively under the same conditions until the decanted fluid is clear. The time first selected should correspond to a computed diameter of 10 microns. The decanted fluid may be filtered through a glass bottom gooch or alundum crucible, dried and weighed, or it may be discarded and estimated by difference.

A new time is selected to correspond to the fall of 20 micron particles through the decantation height. The run is again made in the same manner using this new time, which should be just one-quarter of the time first used. In this case, the decanted fluid may be allowed to settle and any clear supernatant fluid decanted. The concentrate containing all of this fraction is then filtered, dried and weighed. A third run is then made using a time corresponding to the settling of 40 micron particles through the decantation height, and after this both decanted solids and residual solids are filtered, dried, and weighed. A sieve test may then be run on the residual solids, or they may be divided into sieve and sub-sieve ranges by computation from sieve data on another sample.

It is necessary to calculate sieve diameters and Stokes' Law diameters to a common basis. This may be done by microscopic measurement or by microscopic exploration. If the latter method is used, the boundary limits of the coarsest sub-sieve fraction are determined. A small portion is spread on a microscope slide with a drop of water or with diacetone alcohol, the slide is dried and particles are measured under the microscope, with a scale calibrated from a stage micrometer. Either an ocular insert or projection measurement may be used. The small dimension visible in the microscope is used.

REPORT

Measure approximately the coarsest 5% and the finest 10% of the particles in the fraction. Average each separately. Compare the average of the coarsest with the average from the sieve. These should check within 2 to 4 microns and the average of these should be taken as the microscopic diameter corresponding to the sieve cut (74 microns). The value may be as high as 90 to 100 microns, as the diameter measured could pass the sieve aperture on the diagonal. Check the finest size, for this fraction, by measuring the coarsest size of the next finer fraction in the same manner as before. If these two values check within 5 microns, the separation has been satisfactory and the cuts for the smaller fractions may be computed from this average. Each succeeding fraction was so timed that it was cut at twice the diameter of the one before it. Therefore the average measured diameter for the coarsest and next coarsest fraction may be halved to determine the diameter between each succeeding finer fraction. To correlate with sieve diameters, multiply these microscope diameters by the ratio between sieve aperture (74 microns for No. 200) and the microscopic diameter established for this cut. Represent values in the same manner as sieve values, and in connection with them. The per cent on sieve cannot be used unless the intervals chosen for settling correspond to theoretical sieves in the type of nest used; but cumulative and per cent per micron plots connect sieve and settling data properly.

Wet elutriation may be performed in a standard type of apparatus such as the Nobel Elutriator (see Perry, p. 1449). The same type of fractionation results and the product is collected, recovered, and weighed as in the case of beaker sedimentation.

EXPERIMENT NO. 66

Microscopic Measurement

Object. To determine particle size by microscopic measurement.

Microscopic measurement may be used with or without elutriation. If it is used without elutriation, it is not only unnecessary to sieve but it is even undesirable to do so to avoid dust loss. The slide is made with the fresh sample and counted in intervals up to the microscope diameter of the finest sieve cut. The per cent finer than sieve size is then distributed on the basis of the count. For the procedure refer to A.S.T.M. Standards (Part III, pp. 1122-1126 [1939]). It should be noted that it is difficult to prepare a representative slide for measurement over the range from 0-74 microns sieve diameter, and it is preferable to elutriate the sample into at least two fractions in this region, as 10-74 and 0-10 microns.

EXPERIMENT NO. 67

Turbidimetric Measurement

Object. To determine particle size by turbidimetric measurement.

Turbidimetric measurement of surface may be made with the Wagner turbidimeter previously noted. Relative measurements of turbidity may be made with the simpler Jackson or Parr turbidimeters. Weigh out .200 gram of sample, disperse with a small amount of water containing ammonia or saponin, dilute to 100 cc., and read the depth at which the light image is obscured. Dilute the entire sample progressively to 200, 400, 800, and if need be to 1600 cc. and make similar observations. A plot of depth against dilution usually yields a straight line intersecting the depth axis slightly above zero. If the line is not straight, its slope may be roughly estimated, but the surface measurement is correspondingly less exact. Assume a fixed depth, say 12 cm., and note dilutions for each sample of ground material of one kind (or index) of refraction. These dilutions indicate the extension of hiding power with grinding and are an indication of surface.

GENERAL CONSIDERATIONS FOR EXPERIMENTS NO. 68 AND NO. 69 ON GRINDABILITY

Object. 1. To determine grindability of several typical substances. 2. To measure performance characteristics and fineness distribution of products on several types of grinding mills, using a homogeneous and a heterogeneous material as mill feed.

3. To compare ball mill operation
    a. As a function of time, loading, and fluid medium.
    b. As affected by addition agents.

Equipment.

Grindability Tester

Hardgrove machine or ball mill.

Mills

- Jaw crusher.
- Crushing rolls.
- Disc mill.
- Hammer mill.
- Ball mill.
- Screen separator.

Analytical Equipment

- Standard sieves ($\sqrt{2}$ series).
- Sieve shaking machines.
- Pan balance and weights.
- Sample bottles.
- Riffle sampler.
- Oilcloth for quartering samples.
- Shovels and scoops.
- Microscope and accessories.
- Elutriator.
- Turbidimeter.
- General laboratory equipment, such as beakers, burners, etc.

Procedure. *Grindability Test:* Following one of the procedures given here in the authorized reprint from the Proceedings of the American Society for Testing Materials, determine grindabilities for samples designated by instructor. The following list is suggested from which three or four may be chosen:

- Barytes
- Bauxite
- Calcite
- Cement clinker
- Coal, anthracite
- Coal, bituminous
- Dolomite
- Feldspar
- Fused aluminum oxide
- Glass
- Granite
- Gypsum
- Limestone
- Mica schist
- Phosphate rock
- Quartz
- Rock salt
- Talc
- Trap rock

EXPERIMENT NO. 68

TENTATIVE METHOD OF TEST FOR GRINDABILITY OF COAL BY THE BALL-MILL METHOD[1]

Object. To determine the relative amounts of energy necessary to pulverize different coals.

This method describes a laboratory procedure for estimating the grindability of coal. Briefly, the relative amounts of energy necessary are determined by placing a sample of coal in a ball mill and finding the numbers of revolutions required to grind it so that 80% of the sample passes a 74-micron (No. 200) A.S.T.M. sieve.

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[1] This is a Tentative Standard and under the Regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1935; Revised, 1937.

Equipment. The apparatus for the grindability test shall consist of the following:

Ball Mill. A cylindrical steel mill 8 inches in length and 8⅛ inches in diameter, inside dimensions, and ¼ inch in thickness shall be used. Three steel lifters, each $\frac{7}{16}$ inch square and 8 inches in length, shall be riveted longitudinally at 120-degree intervals to the inside wall of the mill. They shall be so fastened that no space is left between the wall and the lifters. The lid shall be fitted to the mill by means of a rubber gasket and held securely in place by the yoke and crossbar arrangement ordinarily employed with laboratory porcelain jar mills. A suitable mill can be made of a section of ordinary 8-inch water pipe with a bottom and lid of ¼-inch steel plate. The bottom may be welded to the pipe section.

Ball Charge. The charge for the mill shall consist of 100 steel ball bearings, each 1 inch in diameter.

Note. Ball bearings are specified because they are of uniform diameters and smoothly polished.

Rotation of Mill. The mill shall be rotated in a horizontal position about its cylindrical axis at 40 r.p.m. The jar-mill frame used for pulverizing coal samples for analysis is suitable for this purpose. A revolution-counter shall be used to show the number of revolutions of the mill.

Sieves. The following sieves will be required: 1680-micron (No. 12), 8 inches or more in diameter, and 74-micron (No. 200), 8 inches in diameter for machine sieving and 10 inches in diameter for hand sieving. The sieves shall conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.

Gross Sample. A representative gross sample of coal shall be collected and prepared in accordance with the Standard Method of Sampling Coal (A.S.T.M. Designation: D 21) of the American Society for Testing Materials. Approximately 10 pounds of coal crushed to pass a 4760-micron (No. 4) sieve shall constitute the laboratory sample.

Procedure. 1. (a) The 10-pound laboratory sample shall be stage-crushed by smoothfaced double rolls or other suitable crusher [Paragraph (b)] in such a manner as to minimize the production of fine sizes. If rolls are used, the 10-pound sample shall be passed in a thin stream through a laboratory-type double-roll crusher set with a spacing of ¼ inch between the rolls. The sample shall then be divided into two equal parts by riffling and one part discarded. The remaining 5-pound portion shall then be screened on the 1680-micron (No. 12) sieve and the oversize passed through the crusher with the rolls set $\frac{3}{16}$ inch apart. The crushed product shall be again screened on the 1680-micron (No. 12) sieve and the undersize combined with that previously obtained. The oversize now remaining on the 1680-micron (No. 12) sieve shall be reduced in size by passing it successively through the crusher with the clearance between the rolls set at $\frac{1}{8}$, $\frac{1}{16}$, and $\frac{1}{32}$ inch, respectively. Material finer than the 1680-micron (No. 12) sieve shall be removed after each crushing, and only the oversize shall be crushed to the next closer roll setting. The 5-pound lot shall then be thoroughly mixed and divided by riffling into two equal parts, one of which shall be set aside as a reserve sample. The other part shall be air-dried at room temperature to constant weight and then screened on the 74-micron (No. 200) sieve to remove the undersize, which shall be discarded. Usually, this undersize product should not exceed 5%.

(b) If other types of laboratory crushers are used, the 10-pound laboratory sample shall be treated as described in Paragraph (a) except as to the type of crusher.

2. (a) A 500-g. sample of the air-dried coal sized between the 1680-micron (No. 12) and 74-micron (No. 200) sieves (Section 4) shall be obtained by riffling. The charge of 100 balls shall be placed in the mill and the 500-g. sample added. Then the mill shall be closed and rotated for 50 revolutions at a speed of 40 r.p.m. The contents of the mill shall then be poured onto a sieve coarse enough to retain the balls but not the coal. The mill and balls shall be brushed to remove the coal. The sample shall be sieved, using either the hand sieving or the mechanical sieving method.

(b) For hand sieving, the product shall be placed on a 10-inch, 74-micron (No. 200) sieve and shaken until not more than 0.5 g. passes in 1 minute of sieving. Certain coals of a sticky nature are difficult to sieve. With such coals, hand sieving is best done by rocking the sieve back and forth briskly on a table top to break up agglomerates and to prevent clogging of the openings.

(c) For mechanical sieving, the product of the ball mill shall be divided into two or more approximately equal parts and each part shall be placed on an 8-inch 74-micron (No. 200) sieve, each provided with a pan. The sieves shall then be shaken for 10 minutes in the sieving machine, after which the sieves shall be removed from the pan and the underside of each carefully cleaned with a 1-inch bristle brush in such a manner that any coal removed falls into the pan. The sieves and pans shall then be returned to the sieving machine for an additional 5-minute period of sieving.

(d) After sieving, the total undersize shall be weighed and the number of revolutions of the mill required to increase the weight of the undersize to 50 g. shall be calculated by direct proportion. This number of revolutions constitutes a "cycle." Both the oversize and the undersize shall be returned to the mill and it shall be rotated for the additional number of revolutions required to complete the cycle. Again the mill shall be removed from the frame and the contents sieved on the 74-micron (No. 200) sieve as before. The undersize product, including the loss, which usually ranges from 1 to 3 g. and is assumed to belong to the undersize, shall weigh between 45 and 55 g. These operations comprise the first cycle. For the second cycle the oversize only shall be returned to the mill and ground for the number of revolutions previously found necessary to give approximately 10% of undersize. The material finer than the 74-micron (No. 200) sieve shall be removed as before. The same procedure of alternate milling of the oversize and removal of the undersize shall be continued with the same number of revolutions per cycle until at least 80% or 400 g. of the original sample passes the 74-micron (No. 200) sieve. The end point of the test shall be considered as the number of revolutions of the mill required to grind exactly 80% of the original sample to pass the 74-micron (No. 200) sieve. This exact number of revolutions shall be determined by interpolation.

Number of Tests. Duplicate tests shall be made. In the second test the preliminary step taken in the first test to determine the number of revolutions necessary to give 50 g. of undersize shall be omitted. The number of revolutions per cycle shall be the same in both tests.

Reproducibility of Results. In duplicate tests made in the same laboratory the number of revolutions required shall agree within 4%.

REPORT

Results shall be expressed as the average number of revolutions required to grind 80% of the sample to pass the 74-micron (No. 200) sieve or as follows:

$$\begin{array}{c}\text{Ball-mill grindability} \\ \text{index, per cent}\end{array} = \frac{50{,}000}{\text{Average number of revolutions}}$$

Rapid Control Modification. For control work on coals having grinding characteristics that have been determined by complete tests, or for large-scale investigations, it shall be considered permissible to shorten the method by taking the end point as the number of revolutions required to grind 40% or 200 g. of the original sample to pass the 74-micron (No. 200) sieve. When the shortened procedure is used the results shall be expressed in terms of 80% or 400 g. passing the 74-micron (No. 200) sieve by extrapolation from the number of revolutions of the mill required to grind 40% of the sample to pass the 74-micron (No. 200) sieve. Extrapolation shall be made by the following formula:

$$R = (2 + X)N + Y$$

where N = the number of revolutions of the mill required to grind 40% of the sample to pass the 74-micron (No. 200 sieve), and
R = the calculated number of revolutions required to grind 80% of the sample to pass the 74-micron (No. 200) sieve.

The proper values of X and Y to give the correct value of R shall be determined by the operator's knowledge of the grinding characteristics of the coal. For many coals X has the value 0.055 and Y equals 47.[1]

Results of shortened tests shall be clearly designated "Ball-mill grindability index by rapid control modification."

APPENDIX

Approximate conversions of ball-mill grindability indexes into Hardgrove grindability indexes, as determined by the Tentative Method of Test for Grindability of Coal by the Hardgrove-Machine Method (A.S.T.M. Designation: D 409-37 T) of the American Society for Testing Materials, may be made by means of a curve plotted from equivalent values, as follows:

Ball-Mill Grindability Index, Per Cent	Equivalent Hardgrove Grindability Index	Ball-Mill Grindability Index, Per Cent	Equivalent Hardgrove Grindability Index
20	29	70	90
30	43	80	100
40	56	90	110
50	68	100	118
60	80		

According to available data, this conversion of ball-mill grindability indexes into Hardgrove grindability indexes should be accurate to approximately ± 3 units for coals having ball-mill grindability indexes ranging from 25 to 50. Sufficient data are not available to determine the accuracy of conversion for coals falling outside this range.

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[1] For information concerning these constants, see discussion by H. L. Brunjes of paper on "Further Investigation of Methods for Estimating the Grindability of Coal," by H. F. Yancey and M. R. Geer, Transactions, Am. Inst. Mining and Metallurgical Engrs., Vol. 119, p..371 (1936).

EXPERIMENT NO. 69

TENTATIVE METHOD OF TEST FOR GRINDABILITY OF COAL BY THE HARDGROVE-MACHINE METHOD[1]

A.S.T.M. Designation: D 409-37 T

Object. This method is used to determine the relative grindability or ease of pulverizing of coals in comparison with a coal chosen as 100 grindability. The method is based on Rittinger's Law, which states: "The work done in pulverizing is proportional to the new surface produced." A prepared sample receives a definite amount of grinding energy in a miniature pulverizer and the new surface is determined by sieving.

Equipment. The apparatus for the grindability test shall consist of the following:

(a) *Grindability Machine.* A grindability machine such as is shown in Fig. 4 is required for this test. The eight 1-inch balls roll on a stationary ring and are driven from above by a rotating ring. The action of the rolling balls causes an increase in the surface of the sample being tested. A definite pressure of 64 pounds ± ½ pound on the balls is obtained by the weights, shaft, top grinding ring, and gear. A predetermining counter is used to stop the motor automatically as soon as the vertical shaft of the grindability machine has made exactly 60 revolutions.

(b) *Sieves.* The following sieves will be required: 1190-micron (No. 16), 590-micron (No. 30), and 74-micron (No. 200). The sieves shall conform to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.

(c) *Mechanical Sieving Device.* A mechanical sieving device is desirable, although not necessary. If a mechanical sieving device is not available, it will be necessary to give the sample an equivalent amount of hand sieving.

(d) *Balance.* A triple beam balance having a sensitivity of 1 cg., and with which weighings from 1 cg. to 111 g. can be made, is well adapted for this test. However, a torsion or trip balance with suitable pan, and of the specified sensitivity may be used. Greater sensitivity than 1 cg. is not necessary for this test.

(e) *Laboratory Crusher.* The sizing of the sample to be placed in the grindability machine shall be such that it will pass the 1190-micron

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[1] This is a Tentative Standard and under the regulations of the Society is subject to annual revision. Suggestions for revision should be addressed to the Headquarters of the Society, 260 S. Broad St., Philadelphia, Pa.

Issued, 1935; Revised, 1937.

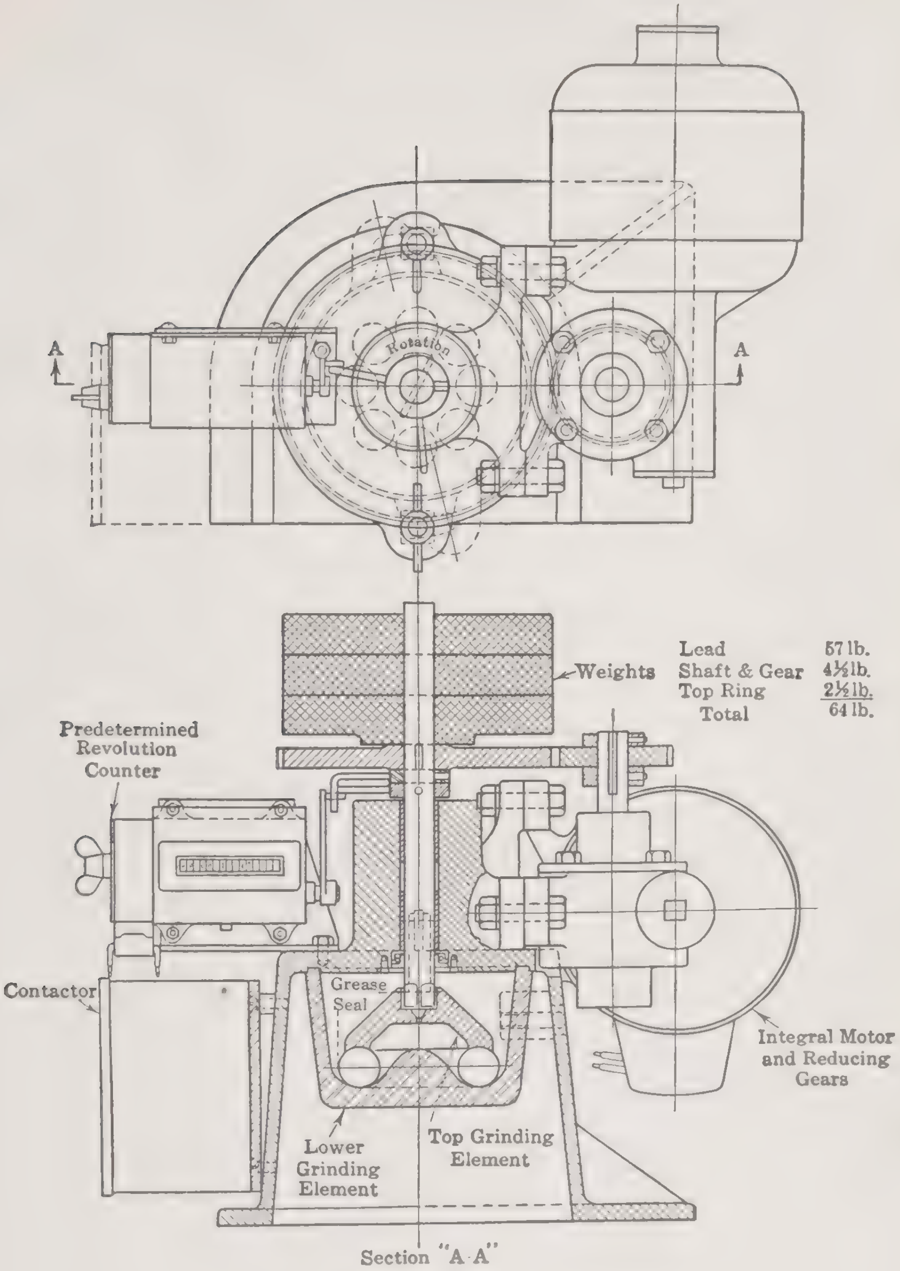
A
Rotation
A
Weights
Lead 57 lb.
Shaft & Gear 4½ lb.
Top Ring 2½ lb.
Total 64 lb.
Predetermined Revolution Counter
Contactor
Grease Seal
Integral Motor and Reducing Gears
Lower Grinding Element
Top Grinding Element
Section "A-A"

Fig. 4.

(No. 16) sieve, and remain on the 590-micron (No. 30) sieve. In order to break up the coarser particles of the sample being prepared for test, a laboratory crusher or a coffee mill will be found desirable. The use of such a device allows the reduction of the coarser particles without the production of excess fine material.

GROSS SAMPLE. A representative gross sample of coal shall be collected and prepared in accordance with the Standard Method of Sampling Coal (A.S.T.M. Designation: D 21) of the American Society for Testing Materials.

Procedure. PREPARATION OF SAMPLE. An air drier and a riffle sampler, as described in Section 1 (a) and (h), respectively, of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (A.S.T.M. Designation: D 271) of the American Society for Testing Materials, or equivalent are desirable in preparing the sample. The laboratory sample, after air drying until the loss in weight is not more than 0.1% per hour, shall be placed on the 1190-micron (No. 16) sieve nested with a 590-micron (No. 30) sieve and bottom pan. The sieves shall be shaken by a mechanical sieving machine for approximately 2 minutes, or given an equivalent amount of hand sieving. The material remaining on the 1190-micron (No. 16) sieve shall be put into a laboratory crusher or a coffee mill in which the set screw has been adjusted so that the coarsest particles are broken up. The sample after passing through the laboratory crusher or coffee mill shall be returned to the 1190-micron (No. 16) sieve of the nest of sieves and again shaken by the mechanical sieving machine or hand sieved. This operation shall be repeated several times until the entire sample has passed through the 1190-micron (No. 16) sieve. The material remaining on the 590-micron (No. 30) sieve shall then be shaken to thoroughly remove any material smaller than this size. All material passing the 590-micron (No. 30) sieve shall be discarded. By using this method of preparing the sample, a minimum of fines is produced.

OPERATION OF GRINDABILITY MACHINE. The predetermined counter shall be set so that the machine will automatically stop after 60 revolutions have been completed. To insure stoppage of the machine within one-quarter of one revolution, two trippers are furnished. One tripper is fixed, and the other may be set to correspond with the coasting of the machine after the switch is opened. Therefore, the number of counts on the counter will be 120, or just twice the number of revolutions made by the machine. The grinding elements shall be thoroughly cleaned by brushing before the sample to be tested is added. The eight 1-inch diameter balls shall be placed in the bottom of the lower grinding element and 50 g. of the prepared No. 16 to No. 30 size sample shall be distributed evenly over the balls. Any material falling on the elevated section of the lower grinding element shall be brushed toward the balls.

The upper grinding element shall then be placed on the balls and the lower end of the shaft shall be fitted to the square opening in the disc and the grinding elements securely fastened in place by two thumb screws on the sides of the lower grinding element. Care shall be taken to pull the bowl up evenly with the thumb screws. The set screw on the collar which supports the driving gears shall always be kept tight, otherwise the weights will rest on the frame and there will be no pressure on the balls. The predetermining counter shall be set to zero, and the starting switch closed. After the machine has been automatically stopped, the sample shall be transferred to the 74-micron (No. 200) sieve.

SIEVING. The 74-micron (No. 200) sieve shall be shaken by a mechanical sieving machine for 10 minutes, or given an equivalent amount of hand sieving. The sieve shall then be removed and the underside carefully cleaned with a 1-inch bristle brush in order to remove any adherent material. The sample shall then be shaken by the mechanical sieving machine for 5 minutes more or given an equivalent amount of hand sieving, after which the brushing of the underside of the sieve shall be repeated. After an additional 5 minutes of sieving, the material is ready for weighing. A total of 20 minutes of mechanical sieving, or an equivalent amount of hand sieving, should result in a comparatively clean sieve.

WEIGHING THE SAMPLE. Coals having a high residual moisture content (this is especially true of lignites) lose some of their residual moisture when in a pulverized condition. For this reason rapid and fairly accurate weighing is essential. The material passing through the 74-micron (No. 200) sieve shall be discarded and the material retained on the sieve shall be weighed to within 0.1 g. and the weight recorded.

REPORT

The grindability index shall be calculated from the following equation:

$$\text{Hardgrove grindability index}^{1} = 13 \pm 6.93W$$

where W = the weight of material passing the 74-micron (No. 200) sieve, determined from the weight of the original sample (50 g.) minus the weight of the material retained on the 74-micron (No. 200) sieve.

REPRODUCIBILITY OF RESULTS. The permissible variation between two or more determinations shall not exceed the following:

	Per Cent
Same laboratory	2
Different laboratories	3

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[1] The calibration of the machine may be checked by running samples of low volatile run-of-mine bituminous coal from the Jerome Mine, Upper Kittanning Bed, Somerset County, Pa. The average of three tests should give a Hardgrove grindability index of 100 ± 2.

APPENDIX

Approximate conversions of Hardgrove grindability indexes into ball-mill grindability indexes, as determined by the Tentative Method of Test for Grindability of Coal by the Ball-Mill Method (A.S.T.M. Designation: D 408-37 T) of the American Society for Testing Materials, may be made by means of a curve plotted from equivalent values, as follows:

Hardgrove Grindability Index	Equivalent Ball-Mill Grindability Index, Per Cent	Hardgrove Grindability Index	Equivalent Ball-Mill Grindability Index, Per Cent
20	14	70	52
30	21	80	60
40	28	90	70
50	36	100	80
60	44	110	90

According to available data, this conversion of Hardgrove grindability indexes into ball-mill grindability indexes should be accurate to approximately ± 3 units for coals having Hardgrove grindability indexes ranging from 35 to 70. Sufficient data are not available to determine the accuracy of conversion for coals falling outside this range.

EXPERIMENT NO. 70

Grinding Mill Performance

Size Distribution as a Function of Material. Select a number of the raw materials for test, including heterogeneous materials as mica schist, or granite, crystalline materials of easy cleavage as gypsum or galena, and essentially homogeneous materials as quartz, glass, or anthracite coal. Crush to secure about five pounds of each between No. 4 and one inch sieves.

Procedure. Set the jaw crusher or rolls so that little material coarser than No. 4 sieve results when this feed is passed through the mill. Pass each material through the mill once; feeding steadily but without choking of the mill. Note time required for a weighed amount of each material to pass through the mill, as an indication of ease of grinding. Sieve a representative sample on a nest of sieves having apertures with change as the square root of two (A.S.A. Standard). Plot the logarithm of the per cent on each sieve against the logarithm of sieve aperture. Note that a square root of two nesting gives even intervals on a logarithmic plot. Interpret the curves, taking note of the peak (percentage and diameter), the extent and slope of any straight line relation, and the presence of a predominant grain size, stating its average diameter.

MILL PERFORMANCE TESTS

Select three different types of materials for mill test. Prepare 50-pound lots of each ranging from 1/4 inch to 1 inch in size. Devise flow sheet for crushing and screening so that this material may be fed in uniform sizes to the available mills.

	Feed Size
Jaw crusher	¼ in. to 1 in.
Rolls	No. 4—No. 10 sieve
Disc mills	No. 10—No. 20 sieve
Hammer mills	No. 10—No. 20 sieve
Ball mill	No. 20—No. 40 sieve

With one setting for each mill, just smaller than the minimum size of feed, pass the material through once. Feed at a uniform rate just rapidly enough to keep the mill operating under load, but not so rapidly as to cause crowding or interference. Note time and weight of charge as an indication of mill capacity for each material for a given reduction. If possible to note power, read for each mill, running empty and under load. Using suitable sampling procedures, take samples for analytical sievings. Run size distribution of feed and product with the analytical nest of sieves. Plot cumulative per cent against reciprocal of sieve aperture. Also plot as per cent per micron against logarithm of diameter. For the ball mill, use either one-gallon laboratory jar mill or a larger unit. The weight of charge should be so selected as to fill the voids between the balls. The mill should be between one-third and two-thirds full of balls. Run the mill for a period of eight hours. If a large mill is used, small samples may be withdrawn at the end of one hour, three hours, and eight hours. If a small mill is used, the amount of sample withdrawn may alter the weight of charge by too great an amount. It is better to start three mills with the same number and weight of balls in each and with the same weight of charge, stopping one at each of the specified times and using the entire charge as sample. The mills may be operated with dry charge or with water added as instructed. Run sieve tests on feed and on products. The large amount of material below sieve size with the ball mills makes necessary a sizing procedure by other methods. The following may be used:

a. Elutriation and Microscopic Measurement.
b. Sedimentation Method.
c. Turbidimeter.

Report ball mill data in accordance with instructions for methods used.

BALL MILL STUDIES. A series of experiments is proposed for the comparison of ball mill performance in which operating characteristics are changed but the feed material remains the same. Measurement of turbidity will suffice for the comparison.

Select a material for test, such as trap rock, phosphate rock, or other homogeneous insoluble material, and prepare 10 pounds of feed between No. 20 and No. 40 sieves. Prepare four laboratory jar mills for use in the same rack. Make runs as listed and report relative turbidities. Plot as instructed in any one case.

1. *Function of Time and Charge.*

   Using the same number and weight of balls in two mills, weigh charge into one to just fill the voids in the balls. Weigh one-half that amount of charge into the other mill. Using half the number and weight of balls in another mill, add the original full weight charge. In the fourth mill use the same volume of balls as in the first two mills but change the size or the material, and use the full weight charge. Run the four mills together in the rack, stopping them at stated intervals to remove a weighed sample in amount equal to 2% of the original weight of charge. Suggested times are ½, 1, 2, 4, 8, and 16 hours. Run turbidimeter and express surface in relative or absolute terms. For each mill, plot a curve of turbidity against time at which the sample was taken. Interpret the shapes and relative positions of the curves.

2. *As a Function of Fluid Medium.*

   Using the same four mills with the same number and weight of balls in each, add to one a dry charge which just fills the voids, to another a charge of the same feed equal to one-quarter of that weight, to another the quarter weight charge and a measured volume of water to cover the balls, and to the fourth the quarter weight charge and a volume of lubricating oil equal to the volume of water. Grind each charge for 4 hours, remove from mill, filter off fluid and in the case of oil wash with kerosene and then carbon tetrachloride, then dry, break up, and run sieve tests and turbidities. Interpret the effect of fluid medium on reduction of coarse sizes and production of fines.

3. *Effect of Addition Agents.*

   Using the same four mills with the same number and weight of balls, prepare two with the full weight charge and two with the quarter weight charge and water. To one of the full weight charges add turkey red oil in amount equal to 1% of the weight of charge. To one of the quarter weight wet mixtures add saponin equal to 1% of the dry charge. Run four hours in the mill rack and remove. Filter the wet charges and dry them, and crush the lumps lightly for sampling. Run turbidimeter. Report observations and interpret the effects of addition agents in wet and dry grinding.

HAMMER MILL STUDIES. A series of experiments is proposed for the high-speed hammer mill which is now available in laboratory sizes as a variable speed mill and with different screens for retaining different sizes of material in the mill. A soft stone should be selected, as limestone, not calcite, and an appropriate feed size (No. 8 to No. 20 sieves), with 2 to 5 pounds for each test. Make runs as indicated and measure sieve fineness, turbidity, or, if instructed, sedimentation fineness.

1. *Function of Screen.*

   Set mill with screen approximately the size of feed. Under the high rotational speeds of the mill the feed will not pass through these openings, but will be subject to considerable grinding, before it is sufficiently small to pass out of the mill. Note time of running charge through the mill and any available power indications of the mill. Collect sample, sieve and run through fineness determinations. Note the coarsest size of the product and the general state of subdivision. Repeat using other screens, keeping the amount and size of feed and the mill speed constant.

2. *Variable Mill Speed.*

   Following the procedure outlined above, operate the mill in successive runs at different speeds as directed by the instructor. Record mill speed, time and power indications for the passage of a stated amount and feed. Keep screen and feed size constant. Run sieve and other fineness determinations as instructed and note maximum size of particle as well as other fineness indications, as a function of mill speed.

REPORT

1. Select a milling procedure for grinding 1000 pounds of anthracite culm per hour from one-quarter inch to a fineness of 80% through No. 200 sieve.
2. Show how the Gates crushing surface diagram measures surface, with mathematical proof.
3. Show mathematically that the area under a curve of per cent by weight in each micron of diameter against logarithm of diameter in microns represents surface.
4. Discuss the relative merits of Kick versus Rittinger.
5. Show typical curves for ground products using (cf. Work, A.S.M.E. Buffalo, 1932):
   a. Logarithm of per cent on sieve versus logarithm of diameter.
   b. Per cent per micron versus diameter.
   c. Logarithm of particle frequency versus diameter.
6. Discuss the effect of closed circuit grinding compared with direct grinding on fineness characteristics and operating costs for a given maximum size.

EMULSIFICATION

EXPERIMENT NO. 71

Producing an Emulsion[1]

REFERENCE

Thomas: Colloid Chemistry.

The problem of treating two liquid phases to produce an emulsion bears some relation to the grinding problem in that the mechanical action is often analogous to that used in grinding. However, even an agitator may produce satisfactory emulsions. The emulsion problem really resolves itself into a matter of the characteristics of the two fluids and the nature of the emulsifying agent which maintains the interface.

Procedure. Use triethanolamine oleate and sodium stearate to emulsify kerosene. The former may be made by stirring together 35 grams of triethanolamine and 70 grams of oleic acid. Dilute with water to secure a solution which pours freely. Calculate the weight of oleate per cc. The latter may be made by dissolving 40 grams of Ivory soap in water to a final known volume. The emulsion is preferably made by using a colloid mill. Clean the mill and test its capacity. Note speed and clearance.

a. Stir together 100 cc. of kerosene and 400 cc. of water, pass it through the colloid mill and allow to stand. Note rapidity with which the mixture settles into two layers. Add 5 grams of triethanolamine oleate and pass through the mill again two or three times. If no oil comes to the surface after the emulsion has been allowed to stand five minutes it is called stable. If not stable, add 5 grams more of the oleate and pass through the mill again. Repeat until a stable emulsion is formed. Try diluting this emulsion with water and with oil, whichever fluid dilutes it without formation of a second layer is the continuous phase.

b. Repeat using 400 cc. kerosene and 100 cc. water.

c. Repeat using the same weights of Ivory soap.

d. Using the same proportions of kerosene, soap, and water as found in (a) to give a stable emulsion, make up 75-100 cc. of emulsion by stirring by hand in a beaker.

Examine all emulsions under the microscope noting particularly any differences between (a) and (d). Report (1) capacity of mill, (2) amount of soap required to stabilize each emulsion, (3) type of each emulsion, (4) relative effectiveness of emulsifying agents, and (5) effectiveness of mill over hand stirring in reducing particle size.

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[1] Adapted from experiment, Chemical Engineering Department, Yale University.

SOLUTION AND PRECIPITATION

EXPERIMENT NO. 72

In order to secure the extremely fine sizes of particles needed for pigment and filler purposes, solution and precipitation is often employed. Examples of this include:

1. Calcium carbonate produced by burning limestone and recarbonating milk of lime with carbon dioxide or sodium carbonate.
2. Lithopone produced by reducing insoluble barium sulphate to the soluble sulphide for reaction with soluble zinc sulphate, followed by calcination to improve crystal size.
3. Zinc oxide or zinc sulphide produced by the action of oxygen or sulphur vapor respectively on zinc vapor.

The laws which control the formation and growth of crystals indicate the importance of seed nuclei, the effect of concentration of reacting solutions, viscosity of the final solution, and solubility and the effect of temperature.

CALCIUM CARBONATE

1. Treat a slurry of calcium hydroxide in a beaker with carbon dioxide until the solution is no longer alkaline to litmus. Runs may be made at room temperature and at 50-75° C. Filter, wash, and dry; and then compare the products under the microscope and with the turbidimeter.
2. Mix stoichiometric amounts of 10-15% solutions of calcium chloride and of sodium carbonate. Observe difference between reaction at room temperature and at 75° C. Allow slurries to stand for one hour and note any change, including volume of settled solid and the clarity of supernatant liquid. Filter, wash, dry; and compare under the microscope and in the turbidimeter.

ZINC SULPHIDE

Precipitate zinc sulphide by mixing stoichiometric quantities of approximately molar solutions of zinc sulphate and sodium sulphide at room temperature. Repeat after having first adjusted *p*H of the two solutions to 3.5-4.5. Filter, wash, and divide the moist cake from each precipitate between three crucibles. Dry one at 105° C., calcine another for one hour at 450° C., and calcine the third for one hour at 650° C. Run turbidities on all six products and compare microscopically. Note that turbidities are low on either side of optimum particle size, about .2 micron.

REPORT

1. How does von Weimarn's law apply in these tests?
2. What effect has calcination upon the structure of zinc sulphide?
3. What is the effect of heating the solution upon crystal growth?

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